The advantages of dried and concentrated foods compared to other methods of preservation are described in Chapters 6, 13 and 15. The heat used to dry foods or concentrate liquids by boiling removes water and therefore preserves the food by a reduction in water activity (Chapter 1). However, the heat also causes a loss of sensory characteristics and nutritional qualities. In freeze drying and freeze concentration a similar preservative effect is achieved by reduction in water activity without heating the food, and as a result nutritional qualities and sensory characteristics are better retained. However, both operations are slower than conventional dehydration, evaporation or membrane concentration. Energy costs for refrigeration are high and, in freeze drying, the production of a high vacuum is an additional expense. This, together with a relatively high capital investment, results in high production costs for freeze-dried and freeze-concentrated foods. Nijhuis (1998) has reviewed the relative costs of freeze drying and radio frequency drying (Chapter 18). Freeze drying is the more important operation commercially and is used to dry expensive foods which have delicate aromas or textures (for example coffee, mushrooms, herbs and spices, fruit juices, meat, seafoods, vegetables and complete meals for military rations or expeditions) for which consumers are willing to pay higher prices for superior quality. In addition, microbial cultures for use in food processing (Chapter 7) are freeze dried for long-term storage prior to inoculum generation. Freeze concentration is not widely used in food processing but has found some applications such as pre-concentrating coffee extract prior to freeze drying, increasing the alcohol content of wine and preparation of fruit juices, vinegar and pickle liquors.

### 22.1 Freeze drying (lyophilisation)

The main differences between freeze drying and conventional hot air drying are shown in Table 22.1.
**Table 22.1** Differences between conventional drying and freeze drying

<table>
<thead>
<tr>
<th>Conventional drying</th>
<th>Freeze drying</th>
</tr>
</thead>
<tbody>
<tr>
<td>Successful for easily dried foods (vegetables and grains)</td>
<td>Successful for most foods but limited to those that are difficult to dry by other methods</td>
</tr>
<tr>
<td>Meat generally unsatisfactory</td>
<td>Successful with cooked and raw meats</td>
</tr>
<tr>
<td>Temperature range 37–93°C</td>
<td>Temperatures below freezing point</td>
</tr>
<tr>
<td>Atmospheric pressures</td>
<td>Reduced pressures (27–133 Pa)</td>
</tr>
<tr>
<td>Evaporation of water from surface of food</td>
<td>Sublimation of water from ice front</td>
</tr>
<tr>
<td>Movement of solutes and sometimes case hardening</td>
<td>Minimal solute movement</td>
</tr>
<tr>
<td>Stresses in solid foods cause structural damage and shrinkage</td>
<td>Minimal structural changes or shrinkage</td>
</tr>
<tr>
<td>Slow, incomplete rehydration</td>
<td>Rapid complete rehydration</td>
</tr>
<tr>
<td>Solid or porous dried particles often having a higher density than the original food</td>
<td>Porous dried particles having a lower density than original food</td>
</tr>
<tr>
<td>Odour and flavour frequently abnormal</td>
<td>Odour and flavour usually normal</td>
</tr>
<tr>
<td>Colour frequently darker</td>
<td>Colour usually normal</td>
</tr>
<tr>
<td>Reduced nutritional value</td>
<td>Nutrients largely retained</td>
</tr>
<tr>
<td>Costs generally low</td>
<td>Costs generally high, up to four times those of conventional drying</td>
</tr>
</tbody>
</table>

22.1.1 Theory

The first stage of freeze drying is to freeze the food in conventional freezing equipment. Small pieces of food are frozen rapidly to produce small ice crystals and to reduce damage to the cell structure of the food (Chapter 21). In liquid foods, slow freezing is used to form an ice crystal lattice, which provides channels for the movement of water vapour. The next stage is to remove water during subsequent drying and hence dry the food.

If the water vapour pressure of a food is held below 4.58 Torr (610.5 Pa) and the water is frozen, when the food is heated the solid ice sublimes directly to vapour without melting (Fig. 22.1). The water vapour is continuously removed from the food by keeping the pressure in the freeze drier cabinet below the vapour pressure at the surface of the ice, removing vapour with a vacuum pump and condensing it on refrigeration coils. As drying proceeds a sublimation front moves into the frozen food, leaving partly dried food behind it.

![Fig. 22.1 Phase diagram for water showing sublimation of ice.](image)
The heat needed to drive the sublimation front (the latent heat of sublimation) is either conducted through the food or produced in the food by microwaves (Chapter 18). Water vapour travels out of the food through channels formed by the sublimed ice and is removed. Foods are dried in two stages: first by sublimation to approximately 15% moisture content and then by evaporative drying (desorption) of unfrozen water to 2% moisture content. Desorption is achieved by raising the temperature in the drier to near ambient temperature whilst retaining the low pressure.

In some liquid foods (for example fruit juices and concentrated coffee extract), the formation of a glassy vitreous state on freezing causes difficulties in vapour transfer. Therefore the liquid is either frozen as a foam (vacuum puff freeze drying), or the juice is dried together with the pulp. Both methods produce channels through the food for the vapour to escape. In a third method, frozen juice is ground to produce granules, which both dry faster and allow better control over the particle size of the dried food.

The rate of drying depends mostly on the resistance of the food to heat transfer and to a lesser extent on the resistances to vapour flow (mass transfer) from the sublimation front (Fig. 22.2).

**Rate of heat transfer**

There are three methods of transferring heat to the sublimation front.

1. **Heat transfer through the frozen layer (Fig. 22.2(a)).**
   
   The rate of heat transfer depends on the thickness and thermal conductivity of the ice layer. As drying proceeds, the thickness of the ice is reduced and the rate of heat transfer increases. The heater surface temperature is limited to avoid melting the ice.

2. **Heat transfer through the dried layer (Fig. 22.2(b)).**
   
   The rate of heat transfer to the sublimation front depends on the thickness and area of the food, the thermal conductivity of the dry layer and the temperature difference between the surface of the food and ice front. At a constant cabinet pressure the temperature of the ice front remains constant. These factors are discussed in detail in Chapter 1 and related in equation (1.12). The dried layer of food has a very low thermal conductivity (similar to insulation materials (Chapter 1, Table 1.5)) and therefore offers a high resistance to heat flow. As drying proceeds, this layer becomes thicker and the resistance increases. As in other unit operations, a reduction in the size or thickness of food and an increase in the temperature difference increase the rate of heat transfer. However, in freeze drying, the surface temperature is limited to 40–65°C, to avoid denaturation of proteins and other chemical changes that would reduce the quality of the food.

3. **Heating by microwaves (Fig. 22.2(c)).**
   
   Heat is generated at the ice front, and the rate of heat transfer is not influenced by the thermal conductivity of ice or dry food, or the thickness of the dry layer. However, microwave heating is less easily controlled (Chapter 18) and there is a risk of localised runaway overheating if any ice is melted.

**Rate of mass transfer**

When heat reaches the sublimation front, it raises the temperature and the water vapour pressure of the ice. Vapour then moves through the dried food to a region of low vapour pressure in the drying chamber. 1 g of ice forms 2 m³ of vapour at 67 Pa and, in commercial freeze drying, it is therefore necessary to remove several hundred cubic
metres of vapour per second through the pores in the dry food. The factors that control the water vapour pressure gradient are:

- the pressure in the drying chamber
- the temperature of the vapour condenser, both of which should be as low as economically possible
- the temperature of ice at the sublimation front, which should be as high as possible, without melting.

In practice, the lowest economical chamber pressure is approximately 13 Pa and the lowest condenser temperature is approximately $-35^\circ C$.

Theoretically the temperature of the ice could be raised to just below the freezing point. However, above a certain critical collapse temperature (Table 22.2) the

![Fig. 22.2](image)

**Fig. 22.2** Heat and moisture transfer during freeze drying: (a) heat transfer through the frozen layer; (b) heat transfer from hot surfaces or radiant heaters through the dry layer; (c) heat generated in the ice by microwaves. The graphs show changes in temperature (- - -) and moisture content (——) along the line A→B→C through each sample.

### Table 22.2  Collapse temperatures for selected foods in freeze drying

<table>
<thead>
<tr>
<th>Food</th>
<th>Collapse temperature ($^\circ C$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coffee extract (25%)</td>
<td>$-20$</td>
</tr>
<tr>
<td>Apple juice (22%)</td>
<td>$-41.5$</td>
</tr>
<tr>
<td>Grape juice (16%)</td>
<td>$-46$</td>
</tr>
<tr>
<td>Tomato</td>
<td>$-41$</td>
</tr>
<tr>
<td>Sweetcorn</td>
<td>$-8$ to $-15$</td>
</tr>
<tr>
<td>Potato</td>
<td>$-12$</td>
</tr>
<tr>
<td>Ice cream</td>
<td>$-31$ to $-33$</td>
</tr>
<tr>
<td>Cheddar cheese</td>
<td>$-24$</td>
</tr>
<tr>
<td>Fish</td>
<td>$-6$ to $-12$</td>
</tr>
<tr>
<td>Beef</td>
<td>$-12$</td>
</tr>
</tbody>
</table>

Adapted from Bellows and King (1972) and Fennema (1996).
concentrated solutes in the food are sufficiently mobile to flow under the forces operating within the food structure. When this occurs, there is an instantaneous irreversible collapse of the food structure, which restricts the rate of vapour transfer and effectively ends the drying operation. In practice, there is therefore a maximum ice temperature, a minimum condenser temperature and a minimum chamber pressure, and these control the rate of mass transfer.

During drying, the moisture content falls from the initial high level in the frozen zone to a lower level in the dried layer (Fig. 22.2), which depends on the water vapour pressure in the cabinet. When heat is transferred through the dry layer, the relationship between the pressure in the cabinet and the pressure at the ice surface is:

\[ P_i = P_s + \frac{k_d}{b\lambda_s} (\theta_s - \theta_i) \]  

where \( P_i \) (Pa) is the partial pressure of water at the sublimation front, \( P_s \) (Pa) the partial pressure of water at the surface, \( k_d \) (W m\(^{-1}\) K\(^{-1}\)) the thermal conductivity of the dry layer, \( b \) (kg s\(^{-1}\) m\(^{-1}\)) the permeability of the dry layer, \( \lambda_s \) (J kg\(^{-1}\)) the latent heat of sublimation, \( \theta_s \) (°C) the surface temperature and \( \theta_i \) (°C) the temperature at the sublimation front (°C). The factors that control the drying time are described by Karel (1974).

\[ t_d = \frac{x^2 \rho (M_1 - M_2) \lambda_s}{8 k_d (\theta_s - \theta_i)} \]  

where \( t_d \) (s) is the drying time, \( x \) (min) the thickness of food, \( \rho \) (kg m\(^{-3}\)) the bulk density of dry food, \( M_1 \) the initial moisture content and \( M_2 \) the final moisture content in dry layer. Note that drying time is proportional to the square of the food thickness: doubling the thickness will therefore increase the drying time by a factor of four.

**Sample problem 22.1**

Food with an initial moisture content of 400% (dry-weight basis) is poured into 0.5 cm layers in a tray placed in a freeze drier operating at 40 Pa. It is to be dried to 8% moisture (dry-weight basis) at a maximum surface temperature of 55°C. Assuming that the pressure at the ice front remains constant at 78 Pa, calculate (a) the drying time and (b) the drying time if the layer of food is increased to 0.9 cm and dried under similar conditions. (Additional data: the dried food has a thermal conductivity of 0.03 W m\(^{-1}\) K\(^{-1}\), a density of 470 kg m\(^{-3}\), a permeability of 2.4 \times 10\(^{-8}\) kg s\(^{-1}\), and the latent heat of sublimation is 2.95 \times 10\(^3\) kJ kg\(^{-1}\)).

**Solution to Sample problem 22.1**

(a): From equation (22.1),

\[ 78 = 40 + \frac{0.03}{2.4 \times 10^{-8} \times 2.95 \times 10^6} (55 - \theta_i) \]

\[ 78 = 40 + 0.42(55 - \theta_i) \]

Therefore,

\[ \theta_i = -35.7°C \]

From equation (22.2),
22.1.2 Equipment

Freeze driers consist of a vacuum chamber which contains trays to hold the food during drying, and heaters to supply latent heat of sublimation. Refrigeration coils are used to condense the vapours directly to ice (i.e. reverse sublimation). They are fitted with automatic defrosting devices to keep the maximum area of coils free of ice for vapour condensation. This is necessary because the major part of the energy input is used in refrigeration of the condensers, and the economics of freeze drying are therefore determined by the efficiency of the condenser:

\[
\text{efficiency} = \frac{\text{temperature of sublimation}}{\text{refrigerant temperature in the condenser}}
\]

Vacuum pumps remove non-condensable vapours. Different types of drier are characterised by the method used to supply heat to the surface of the food. Conduction and radiation types are used commercially (convection heating is not important in the partial vacuum of the freeze drier cabinet) and microwave freeze drying is also now used. Both batch and continuous versions are found for each type of drier. In batch drying, the product is sealed into the drying chamber, the heater temperature is maintained at 100–120°C for initial drying and then gradually reduced over a drying period of 6–8 hours. The precise drying conditions are determined for individual foods, but the surface temperature of the food does not exceed 60°C. In continuous freeze drying, trays of food enter and leave the drier through vacuum locks. A stack of trays, interspersed by heater plates is moved on guide rails through heating zones in a long vacuum chamber. Heater temperatures and product residence times in each zone are pre-programmed for individual foods, and microprocessors are used to monitor and control process time, temperature and pressure in the chamber, and the temperature at the product surface (also Chapter 2). Further details of freeze drying equipment are given by Lorentzen (1981).

Contact (or conduction) freeze driers

Food is placed onto ribbed trays which rest on heater plates (Fig. 22.3(a)). This type of equipment dries more slowly than other designs because heat is transferred by conduction.

\[
t_d = \frac{(0.005)^2 470(4 - 0.08)2.95 \times 10^6}{8 \times 0.03 [55 - (-35.7)]} = 6238.5 \text{ s} \\
\approx 1.7 \text{ h}
\]

(b): From equation (22.2),

\[
t_d = \frac{(0.009)^2 470(4 - 0.08)2.95 \times 10^6}{8 \times 0.03 [55 - (-35.7)]} = 20224 \text{ s}
\]

\approx 5.6 \text{ h}

Therefore increasing the thickness of the layer of food from 0.5 to 0.9 cm results in an increase of 3.9 h to the drying time.
to only one side of the food. There is uneven contact between the frozen food and the
heated surface, which further reduces the rate of heat transfer. There is also a pressure
drop through the food which results in differences between the drying rates of the top and
bottom layers. The vapour velocity is of the order of 3 m s\(^{-1}\) and fine particles of product
may be carried over in the vapour and lost. However, contact freeze driers have higher
capacity than other types.

**Accelerated freeze driers**
In this equipment, food is held between two layers of expanded metal mesh and subjected
to a slight pressure on both sides (Fig. 22.3(b)). Heating is by conduction, but heat is
transferred more rapidly into food by the mesh than by solid plates, and vapour escapes
more easily from the surface of the food. Both mechanisms cause a reduction in drying
times compared with contact methods.

**Radiation freeze driers**
Infrared radiation from radiant heaters (Chapter 18) is used to heat shallow layers of food
on flat trays (Fig. 22.3(c)). Heating is more uniform than in conduction types, because
surface irregularities on the food have a smaller effect on the rate of heat transfer. There
is no pressure drop through the food and constant drying conditions are therefore created.
Vapour movement is approximately 1 m s\(^{-1}\) and there is little risk of product carryover.
Close contact between the food and heaters is not necessary and flat trays are used, which
are cheaper and easier to clean.

**Microwave and dielectric freeze driers**
Radio frequency heaters have potential use in freeze drying but are not widely used on a
commercial scale. They are difficult to control because water has a higher loss factor than
ice and any local melting of the ice causes ‘runaway’ overheating in a chain reaction (Chapter 18).

A modification of freeze drying is named reversible freeze-dried compression. Food is freeze dried to remove 90% of the moisture and it is then compressed into bars using a pressure of 69 000 kPa. The residual moisture keeps the food elastic during compression, and the food is then vacuum dried. When packaged in inert gas these foods are reported to have a shelf life of five years. They are used in military rations (for example a meal consisting of separate bars of pepperoni, stew, granola dessert and an orange drink). The bars reconstitute rapidly, during which time the compressed food ‘groans, rumbles, quivers and eventually assumes its normal shape and size’ (Unger, 1982).

### 22.1.3 Effect on foods

Freeze-dried foods have a very high retention of sensory characteristics and nutritional qualities and a shelf life of longer than 12 months when correctly packaged. Volatile aroma compounds are not entrained in the water vapour produced by sublimation and are trapped in the food matrix. As a result, aroma retention of 80–100% is possible. Theories of volatile retention are discussed in detail by Karel (1975) and Mellor (1978).

The texture of freeze-dried foods is well maintained; there is little shrinkage and no case hardening (Chapter 15). The open porous structure (Fig. 22.4) allows rapid and full rehydration, but it is fragile and requires protection from mechanical damage. There are only minor changes to proteins, starches or other carbohydrates. However, the open porous structure of the food may allow oxygen to enter and cause oxidative deterioration of lipids. Food is therefore packaged in an inert gas (Chapter 20). Changes in thiamin and ascorbic acid content during freeze drying are moderate and there are negligible losses of other vitamins (Table 22.3). However, losses of nutrients due to preparation procedures,

![Porous structure of freeze-dried food.](image)

**Table 22.3** Vitamin losses during freeze drying

<table>
<thead>
<tr>
<th>Food</th>
<th>Vitamin C (Loss %)</th>
<th>Vitamin A (Loss %)</th>
<th>Thiamin (Loss %)</th>
<th>Riboflavin (Loss %)</th>
<th>Folic acid (Loss %)</th>
<th>Niacin (Loss %)</th>
<th>Pantothenic acid (Loss %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Beans (green)</td>
<td>26–60</td>
<td>0–24</td>
<td>–</td>
<td>0</td>
<td>–</td>
<td>10</td>
<td>–</td>
</tr>
<tr>
<td>Peas</td>
<td>8–30</td>
<td>5</td>
<td>0</td>
<td>–</td>
<td>–</td>
<td>0</td>
<td>10</td>
</tr>
<tr>
<td>Orange juice</td>
<td>3</td>
<td>3–5</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>–</td>
<td>–</td>
</tr>
<tr>
<td>Beef</td>
<td>–</td>
<td>–</td>
<td>2</td>
<td>0</td>
<td>+</td>
<td>0</td>
<td>13</td>
</tr>
<tr>
<td>Pork</td>
<td>–</td>
<td>–</td>
<td>&lt;10</td>
<td>0</td>
<td>–</td>
<td>0</td>
<td>56</td>
</tr>
</tbody>
</table>

*+, apparent increase.*

Adapted from Flink (1982).
especially blanching of vegetables, may substantially affect the final nutritional quality of a freeze-dried food.

22.2 Freeze concentration

Freeze concentration of liquid foods involves the fractional crystallisation of water to ice and subsequent removal of the ice. This is achieved by freezing, followed by mechanical separation techniques (Chapter 6) or washing columns. Freeze concentration comes closest to the ideal of selectively removing water from a food without alteration of other components. In particular, the low temperatures used in the process cause a high retention of volatile aroma compounds. However, the process has high refrigeration costs, high capital costs for equipment required to handle the frozen solids, high operating costs and low production rates, compared with concentration by boiling (Chapter 13). The degree of concentration achieved is higher than in membrane processes (Chapter 6), but lower than concentration by boiling. As a result of these limitations, freeze concentration is only used for high-value juices or extracts (Thijssen, 1982).

22.2.1 Theory

The factors that control the rate of nucleation and ice crystal growth are described in Chapter 21. In freeze concentration it is desirable for ice crystals to grow as large as is economically possible, to reduce the amount of concentrated liquor entrained with the crystals. This is achieved in a paddle crystalliser by slowly stirring a thick slurry of ice crystals and allowing the large crystals to grow at the expense of smaller ones (Muller, 1967). Details of the effect of solute concentration and supercooling on the rate of nucleation and crystal growth are described by Thijssen (1974). Calculations of the degree of solute concentration obtained by a given reduction in the freezing point of a solution are used to produce freezing point curves for different products (Fig. 22.5).

The efficiency of crystal separation from the concentrated liquor is determined by the degree of clumping of the crystals, and amount of liquor entrained. Efficiency of separation is calculated using:

\[ \eta_{sep} = x_{mix} \frac{x_i - x_j}{x_i - x_j} \]

![Fig. 22.5](image.png) Freezing point curves: curve A, coffee extract; curve B, apple juice; curve C, blackcurrant juice; curve D, wine. (After Kessler (1986).)
where $\eta_{\text{sep}}$ (%) = efficiency of separation, $x_{\text{mix}}$ = weight fraction of ice in the frozen mixture before separation, $x_i$ = weight fraction of solids in liquor after freezing, $x_i$ = weight fraction of solids in ice after separation and $x_j$ = weight fraction of juice before freezing.

Separation efficiencies of 50% for centrifuging, 71% for vacuum filtration, 89–95% for filter pressing and 99.5% for wash columns (Section 22.2.2) are reported (Mellor, 1978).

### 22.2.2 Equipment

The basic components of a freeze concentration unit are shown in Fig. 22.6. These are:

- a direct freezing system (for example solid carbon dioxide) or indirect equipment (for example a scraped surface heat exchanger (Chapters 12, 13, 21)) to freeze the liquid food
- a mixing vessel to allow the ice crystals to grow
- a separator to remove the crystals from the concentrated solution.

Separation is achieved by centrifugation, filtration, filter pressing (Chapter 6) or wash columns. Wash columns operate by feeding the ice-concentrate slurry into the bottom of a vertical enclosed cylinder. The majority of the concentrate drains through the crystals and is removed. The ice crystals are melted by a heater at the top of the column and some of the melt water drains through the bed of ice crystals to remove entrained concentrate. Detailed descriptions of wash columns are given by Mellor (1978).

Concentration takes place in either single-stage or, more commonly, multi-stage equipment. Multi-stage concentrators have lower energy consumption and higher production rates. Improvements in techniques for generating large ice crystals and more efficient washing have increased the maximum obtainable concentration to 45% solids (Kessler, 1986). The energy consumption and the degree of concentration achieved by freeze concentration, in comparison with other methods of concentration, are shown in Chapter 13 (Table 13.3).

### 22.3 Acknowledgements

Grateful acknowledgement is made for information supplied by Atlas Industries A/S, DK-2750 Ballerup, Denmark.
22.4 References


