

# 3

## Separation and Concentration Technologies in Food Processing

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### 3.1 Introduction

Separation and concentration technologies are among the most important unit operations in food processing. From disk-bowl centrifugation for industrial-scale production of skim milk to crystallization for sucrose or ultrafiltration to recover soluble proteins from cheese whey, separation and concentration processes have improved food processing. These technologies have allowed the development of new food products and are being increasingly used for water recycling in food processing. Indeed, food processing consumes large volumes of water. For this reason, waste water treatments use membrane technologies as part of the solution to numerous environmental problems posed by the food industries.

The first processes developed to separate food components selected physical or mechanical means that allowed simple separations involving solid–solid or solid–liquid systems. Screening processes separated solid–solid materials into different sizes, while filtration strictly involved the separation of solid–liquid systems. Among screening methods, the pneumatic separator used compressed gases to separate particles according to their masses; the vibration separator relied on vibration to move particles through different screens separating solids by their sizes or masses; and the magnetic separator applied magnetic fields to remove metallic particles. As for the filtration method, it included conventional filtration, mechanical expression (i.e. for juice extraction from fruits or vegetables), centrifugation, and membrane technologies.

Another group of separation and concentration technologies relied on heat-induced phase changes as the driving force for the separation. From simple evaporation to distillation and solvent extraction, such approaches allowed for the concentration of many liquid foods (i.e. milk, fruit and vegetable juices, etc.) and for as the industrial production of ethanol, liquor, and vegetable oils. The most recent development involving phase change is the use of supercritical carbon dioxide (CO<sub>2</sub>), which has found many value-added applications for the food industry over the past decade.

Conventional filtration relies on gravity, pressure or vacuum to create the driving force necessary for the liquid phase to pass throughout different kinds of filters (e.g. perforated plates, cellulose filter papers, glass fiber filters) or granular material (e.g. sand or anthracite). Membrane technologies, including reverse osmosis (RO), nanofiltration (NF), ultrafiltration (UF), and microfiltration (MF), use pressure differences as the driving force of separation. Like conventional filtration, membrane filtration relies on filters but with much smaller pores. It thus covers a wide range of separations from the removal of particles >1 µm (MF) to water purification by the removal of solutes <10<sup>-1</sup> nm (RO) (Cui et al., 2010). In addition, recent developments in electrically driven separations (electrodialysis), low-pressure separation (pervaporation), and separations using functionalized membranes (ion exchange materials) are opening new horizons in this fascinating field. Future trends in separation and concentration technologies will be characterized by an increased number of integrated combinations, or hybrid processes, combining single units of the before mentioned array of technologies

in order to improve energy efficiency and provide environmentally sustainable processes (Muralihara, 2010).

Several challenges must be met in the design of efficient and economical separation and concentration technologies for the food industry.

- **The diversity and complexity of food systems:** each food system has its own physical characteristics whether it is a liquid (viscosity), a suspension (size, shape, concentration of particles) or a solid (mechanical and/or textural properties).
- **The lability of food components during processing conditions:** many food components are prone to chemical changes when exposed to high temperatures, intense shear stress or the presence of oxygen.
- **The need for safe technologies:** the goal of any food processing method is to provide the consumer with nutritious and safe foods, that is, microbiologically clean and free from external contaminants.
- **The need for low-energy, sustainable processes:** the uncertain future of fossil energy, the rising concerns about greenhouse gases and their effects on the population call for new processes that will minimize energy consumption and carbon footprint.

The objectives of this chapter are to provide an overview of separation and concentration technologies used in the food industry and a better understanding of their underlying principles, associated advantages, and limitations. This chapter also introduces new emerging technologies having potential use for the food industry.

### 3.2 Physical separation of food components

Physical separation methods are generally suitable for removing suspended solids from slurries or for separating solid particles of mixtures.

#### 3.2.1 Filtration

Solid–liquid separations can be performed using two approaches: (1) sedimentation, most often by means of gravity, and (2) filtration. In the food industry, sedimentation is used mainly for waste water treatment and will not be covered in this chapter.

Filtration involves the removal of insoluble particles from a suspension by passing it across a porous material, retaining particles according to their sizes – and shape to some extent. As the filter media retain the larger particles and form a “filter cake”, the permeate passes through the

**Table 3.1** Summary of the types of filtration equipment used in food processing

Equipment type	Driving force	Advantages // Limitations
Plate-and-frame filter press	Pressure	Large filtration area, operating at high pressures (up to 25 bar), filter cake easy to remove, high-quality filtration, low capital cost // Batch mode only, automation difficult
Horizontal plate filters		
Shell-and-leaf filters		
Edge filters		
Rotary drum filter	Pressure or vacuum	Continuous filtration, low manpower requirement // Lower filtration area and higher capital cost (especially for drum filters)
Rotary vacuum disk filters		
Centrifugal filters	Centrifugal	Suitable for batch or continuous mode // Complex handling, filter cake (sludge) difficult to remove

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filter barrier. The mean size particles and their distribution will both have a great influence on the type of filter used. Furthermore, the technique chosen will depend on whether the solid or the liquid is of interest to the manufacturer. Both efficiency and cost-effectiveness should be considered in the type of filter chosen. As shown in Table 3.1, pressure, vacuum or centrifugal forces (discussed in section 3.2.2) can all be used as the driving forces for filtration, which can be achieved using membrane filters, disk filters, cartridges, woven wire screens or packed beds made from organic and inorganic materials (i.e. minerals, carbon, glass, metal, and ceramics) (Sutherland, 2008). Filtration aids, such as diatomaceous earth, cellulose or charcoal, are often used as absorbents to control the formation and the properties of the filter cake, in order to prevent resistance to fluid flow.

Filtration equipments are mainly used in edible oil refining, sugar refining, beer production, wine making, and fruit juice processing. The uses of MF and UF (see section 3.4) for those latter applications are increasing, as more efficient industrial equipment and membranes become available. However, compared to conventional

filtration, MF and UF membranes are less inclined to clog and offer greater mechanical strength (e.g. against pressure) and chemical resistance. Furthermore, membrane processes are cleaner, since they avoid the use of filtering aids such as diatomaceous earth (Daufin et al., 2001).

### 3.2.2 Centrifugation (separators, clarifiers)

A centrifuge is a device that separates particles from suspensions, or even macromolecules from solutions, according to their size, shape, and density. This method subjects dispersed systems to artificially induced gravitational fields (i.e. centrifugal force) as shown in Figure 3.1. Centrifugation can also be used for the separations of immiscible liquids. Industrial centrifugation can be divided into two different classes: (1) sedimenting and (2) filtering.

The sedimenting type of centrifuge relies on density (i.e. specific gravity) differences as a driving force to separate the components of a mixture. Indeed, the rotation of

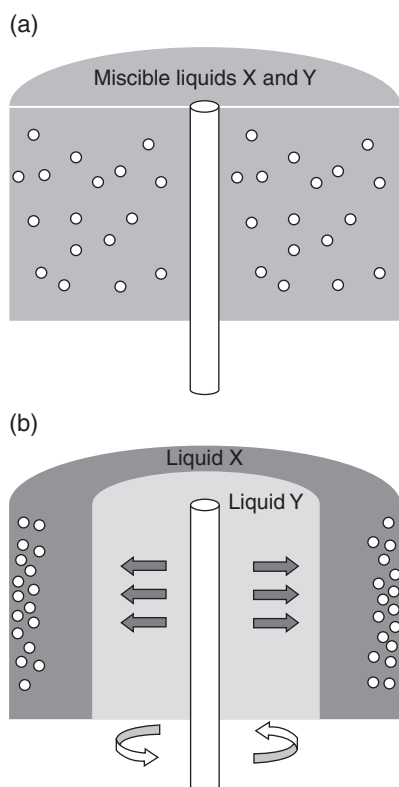
materials around a fixed axis generates a centrifugal force as much as 10,000 times greater than gravity, which acts differently on components depending on their specific gravity. Because denser particles require a greater force to stay close to the rotation axis than lighter particles, the denser constituents are forced to the periphery of the centrifuge bowl. This type of centrifuge is suitable for the separation of solid–liquid and liquid–liquid mixtures with very small gravity differences, as low as  $100 \text{ kg/m}^3$  (Sinnott, 2005).

Filtration centrifuge, in contrast to sedimentation centrifuge, uses perforated bowls and is restricted to the separation of solid–liquid mixtures. This type of centrifugal separation allows the liquid phase to permeate through the porous wall on which solids are retained. Unlike the sedimenting centrifuge, the filtering type of centrifuge has the advantage of allowing the separation of soluble solids without relying on density differences. Although particles must be at least greater than 10 microns for filtration to be practicable (Leung, 2007).

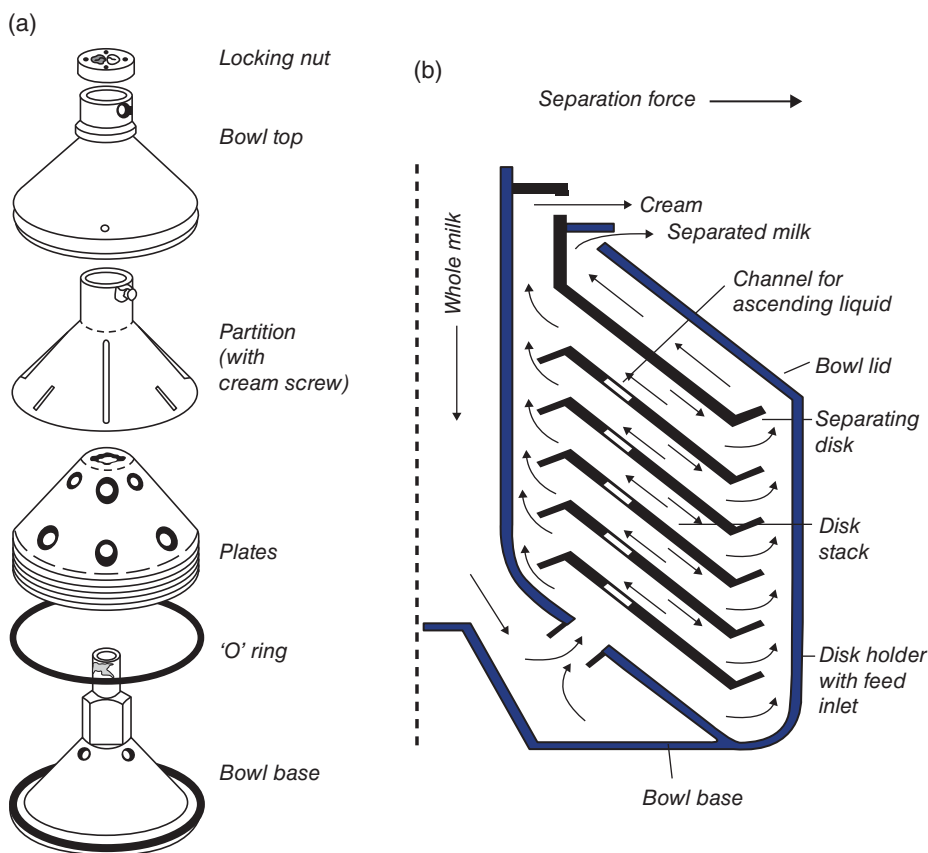
Centrifugation can thus provide solid–liquid and liquid–liquid separations, such as in milk processing, in which milk fat (i.e. cream) removal and clarification can occur simultaneously. Centrifugal clarification is also used to treat oils, juices, and beers. Industrial centrifugation equipment used in food processing plants is mainly of the disk-bowl type, as illustrated in Figure 3.2. In recent years, high-speed centrifugation has been used to reduce bacterial counts in raw milk (i.e. so-called bactofugation) and thus, allow for the production of extended-shelf life (ESL) milk.

### 3.2.3 Pneumatic separation

In the food industry, air is often used to remove foreign particles (e.g. straw, hull, and chaff) or for classification purposes. Pneumatic separation is one of the oldest separation methods known to humankind. This technique allows the standardization of heterogeneous particle mixtures into uniform fractions based on their density and mass. For example, in the grain industry, by using airflows, grain or flours mixtures can be separated according to their size or chemical composition (e.g. protein content) (Vose, 1978). In oil extraction processes, hulls, because of their low density compared to seeds, can be removed using adjusted-force airflows (Kazmi, 2012). Indeed, small particles fall more slowly, have less inertia and can change direction more easily than larger particles. In principle, the smaller particles (e.g. foreign particles) are dragged along with the air stream while larger elements fall through it. The quality of the separation depends on the behaviour



**Figure 3.1** Schematic representation of a mixture of two miscible liquids containing particles: (a) in the absence or (b) in the presence of centrifugal force.



**Figure 3.2** Disk-bowl centrifuge: (a) separator components, (b) separation of cream from milk.

of particles in the air stream. Indeed, each material must possess different aerodynamic properties to be accurately separated. For example, the efficiency of pneumatic separation in flour classification, which refers to the recovery of the desired product from the raw material at a given cut-off point, varies from 50% to 80% (Vose, 1978).

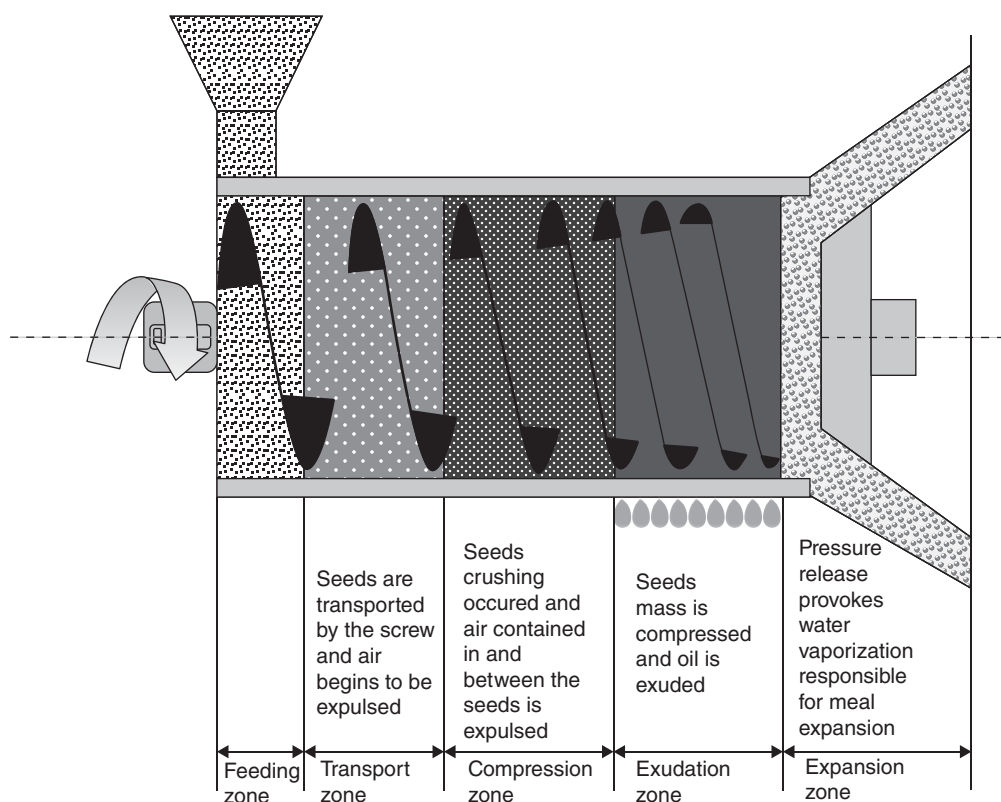
### 3.2.4 Mechanical expression

The extraction of oil or juice from plant materials presents an additional challenge since it requires the disruption of cell structures and hull-protected seeds of varying thickness and resistance. The methods generally used for oil recovery are expression and solvent extraction or the combination of both. Expression is the oldest method applied for oilseeds extraction. The principle of expression is simple. First, the preconditioned oilseeds are pressed in a permeable barrel-like cavity where shear forces, combined or not with heat, squeeze the oil out from the seeds.

Second, the “cake” products, or de-oil material, exit from one side of the press as oil flows throughout the barrel’s openings and is collected (Kazmi, 2012).

Mechanical and thermal pretreatments of seed are both essential to enhance oil extraction process performances. Those treatments generally involve dehulling, flaking or grinding of seed, cooking and sometimes enzymatic pretreatment (Kazmi, 2012; Savoie et al., 2013). As dehulling and reduction size steps ensure homogeneous material and increase the surface area, cooking helps break down oil cells, lowers oil viscosity, adjusts moisture, coagulates proteins, and inactivated enzymes (Kazmi, 2012; Khan & Hanna, 1983). The cooking step is usually done using hot steam. However, it can also use dielectric heat generation (i.e. microwave and radiofrequency) or non-thermal processes such as electric pulsed fields (Kazmi, 2012).

Two kinds of presses can be employed for pressing purposes: the screw press and the hydraulic press. Screw presses have the advantage of giving a higher yield



**Figure 3.3** Schematic representations of the operating zones of a typical screw press (Savoire et al., 2013).

and are suitable for a continuous mode. For this reason, hydraulic presses are only used for specialty oil, olive oil, and for the pressing of cocoa (Savoire et al., 2013). The different operating zones of a common screw press are illustrated in Figure 3.3.

Efficiency of mechanical oil extraction is low compared to solvent extraction. Indeed, depending on the type of seed and the operation parameters (i.e. temperature, pressure, time, and seed moisture content), maximum attainable yield is limited to around 80% (Hasenhuettl, 2005). Moreover, if a yield of 70–80% is generally obtainable for high-fat oilseeds (e.g. sunflowers, sesame, linseed, rapeseed, palm kernel, etc.), the yield drops to roughly 50–70% for seeds containing less than 20% of oil (e.g. soybeans) (Bargale, 1997). However, this process has the advantage of giving high-quality oils, free from dissolved chemical, and is a safer process than solvent extraction (Fellows, 2009). For this reason, expression is generally limited to a small-capacity plant, to high-fat oilseeds (>35%) or to specialty products (e.g. natural or organic oils, essential oils, etc.) (Lin & Koseoglu, 2005; Rosenthal et al., 1996).

In fruit processing, presses can remove most of the juices from the pulp with minimal undesired components (i.e. phenolic compounds which cause bitterness and browning). Using continuous mode, yield of around 84% of juice is obtainable. The efficiency of juice extraction by mechanical expression depends on the maturity of the raw material, the physical resistance of the structure to mechanical deformation, the time, the pressure, the juice viscosity, as well as the temperature (Fellows, 2009).

### 3.3 Processes involving phase separation

#### 3.3.1 Liquid-liquid

##### 3.3.1.1 Crystallization

Crystallization is the process by which solid crystals, of a solute, are formed from a solution (Berk, 2009a). In the food industry, products such as sugar, lactose, glucose, and salt are obtained by this process. It may also be used

to remove undesirable products. For example, edible oils may be cooled to crystallize high melting-point components by a process called winterizing.

Crystallization generally involves four distinctive steps: (1) the generation of a supersaturated state, (2) nucleation, (3) crystal growth, and (4) recrystallization (Hartel, 2001). A supersaturated solution is generated when a solution has reached the solute's maximum concentration and is then further concentrated, usually by evaporation, or cooled down slowly (Hartel, 2001). This first step is the driving force for crystallization by which the system lowers its energy state. Then nucleation takes place as solutes aggregate to form orderly "clusters" or nuclei. There are two kinds of nucleation: (1) homogeneous and (2) heterogeneous. The first occurs without the presence of foreign particles and the second, more common, is aided by the presence of foreign particles in the solution. The subsequent binding of solute molecules to existing nuclei, thus increasing crystal size, is called crystal growth. This reaction stops as an equilibrium state is reached, if the solution is not kept in a supersaturated state by the constant removal of solvent, usually through constant solvent evaporation (Berk, 2009a). Finally, the recrystallization step naturally takes place by the reorganization of the crystalline structure to a low-energy state. The supersaturation ( $\beta$ ), the rate of homogeneous nucleation ( $J$ ), and the rate of crystal growth ( $G$ ), are given by the following equations (Berk, 2009a):

$$\beta = \frac{c}{c_s} \quad (3.1)$$

$$J = a \exp \left[ -\frac{16\pi}{3} \times \frac{V^2 \sigma^3}{(kT)^3 (\ln \beta)^2} \right] \quad (3.2)$$

$$G = k'(C - C_s)^g \quad (3.3)$$

$a$  = a constant

$C$  = concentration (kg solute per kg solvent)

$C_s$  = saturation concentration (kg solute per kg solvent)

$g$  = a numerical called growth order

$k$  = the Boltzmann constant

$k'$  = empirical coefficient

$T$  = temperature, K

$V$  = molar volume of the solute

$\sigma$  = nucleus-solution surface tension

$b$  = supersaturation ratio

The equipment used for crystallisation is called a crystallizer or pan. Pans can crystallize in batch or continuous mode. They can be equipped with a vacuum or be

vented, and generally include a heat exchanger as well as an agitation device.

In the food industry, crystallization is mainly used for sugar refining, most commonly to obtain sucrose. To obtain sucrose, sugar must be removed from sugar canes or sugar beets to produce a diluted liquid which is then clarified to remove impurities (i.e. minerals and organic matters) before further concentration, commonly by evaporation. The sucrose syrup obtained is then subjected to a controlled crystallization process using a vacuum evaporator pan, allowing for the sucrose to separate. Since sucrose solubility in water is high, multiple steps are necessary to remove as much of the sucrose in the solution as possible. Last, sucrose crystals are removed from the mother liquor using centrifugation. This liquor contains a low amount of sucrose and a high concentration of impurities and is further evaporated to obtain molasses (Hartel, 2001).

### 3.3.1.2 Distillation

Distillation is a physical process whereby volatile components of a mixture are separated based on differences in their volatility – the compounds with the lowest boiling point and the highest vapor pressure are separated first. The equilibrium relationships for two-component vapor-liquid mixtures are governed by the relative vapor pressure of its constituents and can conveniently be presented in a boiling temperature-concentration diagram (Figure 3.4). Indeed, the partial vapor of a component ( $P_A$ ) is proportional to its mole fraction ( $X_A$ ) at a specific temperature and the total vapor pressure of a mixture is the sum of the partial pressure of its components ( $P_A + P_B$ ) (Fellows, 2009). This equilibrium relationship can also be expressed mathematically as follows:

$$P_A = X_A \times P_A^\circ \quad (3.4)$$

$$P_B = X_B \times P_B^\circ \quad (3.5)$$

$$\text{Total vapor pressure} = P_A + P_B \quad (3.6)$$

$A$  = component A

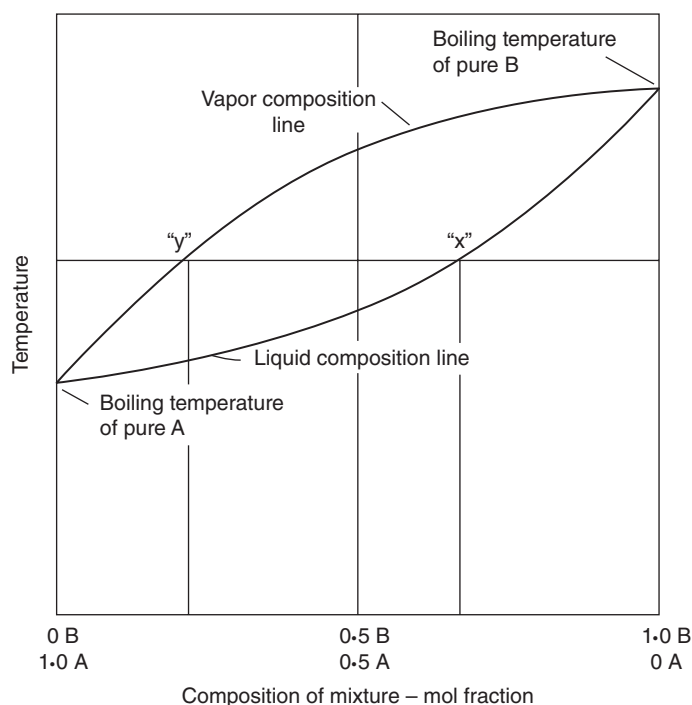
$B$  = component B

$P$  = partial pressure, kPa

$P^\circ$  = partial pressure of the pure component, kPa

$X$  = component fraction, mole of (A or B)/mole total (A + B)

The horizontal line in the boiling temperature-concentration diagram (see Figure 3.4) gives the composition of a boiling liquid (x) and of the vapor (y) at a



**Figure 3.4** Boiling temperature/concentration diagram.

specific temperature. The diagram shows that, at a given temperature, the vapor ( $y$ ) is richer in the more volatile component than the boiling liquid ( $x$ ) and this is the basis for separation by distillation.

For example, in an ethanol–water mixture, as the linkages between similar molecules are greater than between dissimilar ones, following heating, the weaker ethanol–water linkages break down more easily as the more volatile compound (i.e. ethanol) is vaporized. Thereby, after condensation of the vapor, the distillate will be concentrated in stronger ethanol–ethanol linkages and further concentration by vaporization will be harder. In fact, some mixtures, like ethanol–water, form azeotropes in which equilibrium prevents further distillation. Indeed, at 95.6% of ethanol (w/w) and 4.4% of water, the mixture boils at  $78.15^{\circ}\text{C}$ , being more volatile than pure ethanol (i.e. boiling point of  $78.5^{\circ}\text{C}$ ). For this reason, ethanol cannot be completely purified by direct fractional distillation (Fellows, 2009).

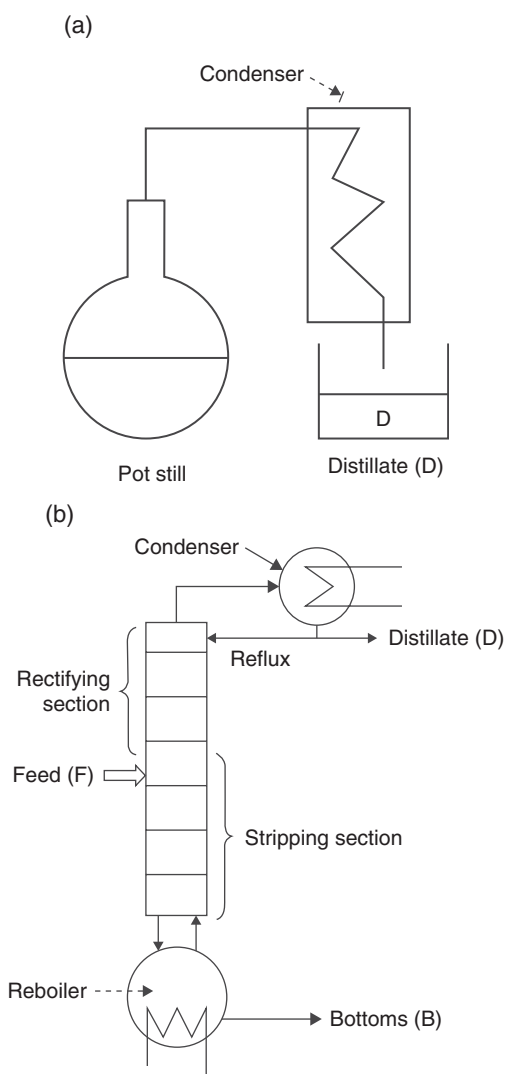
The typical distillation equipment used for continuous fractionation of liquids consists of three main items: (1) a boiler, which generates the necessary heat to vaporize the initial mixture; (2) a column, in which the stages for the distillation separation are provided; and (3) a condenser, for condensation of the final product (i.e. distillate) into the upper column. The distillation column contains multiple

contact stages, through which liquid moves down and vapor moves up (Figure 3.5b). This method allows the vapor traveling up the column to be cooled by the descending liquid and the liquid to be heated by the ascending vapor. The liquid can thus lose its more volatile components as it travels down the column and the vapor can be enriched as it moves up. A part of liquid obtained by condensation of the vapor is fed back (i.e. the reflux) into the upper part of column (i.e. the rectification zone) in order to provide sufficient liquid for contact with the gas (Berk, 2009b).

Even if most industrial distillation operations use continuous distillation columns, distillation in batch mode is still used in the production of spirits such as whiskey and cognac (see Figure 3.5a). In the food industry, despite its simplicity and low capital cost, distillation is mainly confined to the concentration of alcohol beverages, essential oils, volatile flavors, aroma compounds, and to the deodorization of fats and oils. Along with evaporation, distillation is one of the most energy-consuming processes used in the food industry (Fellows, 2009).

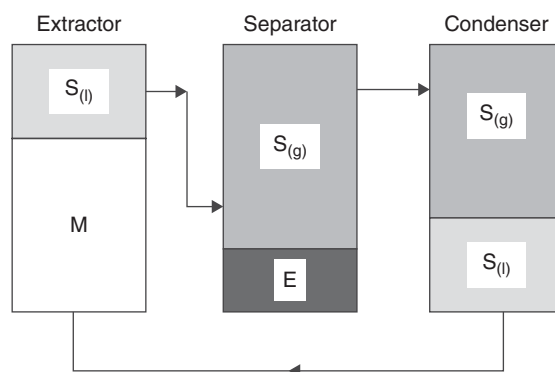
### 3.3.1.3 Solvent extraction

Solvent extraction is the separation of a soluble compound, the solute, by diffusion from a solid (e.g. plant material) or liquid (e.g. oil) matrix using a volatile solvent. For example,



**Figure 3.5** Schematic diagram of a typical distillation apparatus: (a) pot still and (b) distillation columns. Adapted from Berk (2009b).

in the case of compounds extracted from solid materials such as plants, the solid fragments are mixed with solvent and are held for a predetermined lap of time before the removal of the solvent. This holding process involves two stages: (1) an initiation stage; and (2) a diffusion stage. In the initiation stage, solid fragments swell as they absorb solvent and the soluble components are dissolved. Then, diffusion occurs within the fragments outward. The holding time must be sufficient for solutes to dissolve in the



**Figure 3.6** Schematic representation of a solvent extraction unit (S = solvent; M = material; E = extract). Adapted from Grandison and Lewis (1996).

solvent (Fellows, 2009). The fraction extracted is a function of both the distribution ratio ( $D$ ) and the phase ratio ( $\theta$ ) and can be expressed mathematically as follows (Rydberg et al, 2004):

$$D_x = \frac{[X_e]}{[X_a]} \quad (3.7)$$

$$\theta = \frac{C_e}{V_a} \quad (3.8)$$

$$E_x = D_x \theta \div (D_x \theta + 1) \quad (3.9)$$

Where;

$a$  = aqueous phase,  $e$  = solvent,  $x$  = desired component

$D_x$  = distribution ratio

$E_x$  = fraction extracted

$V$  = volume

$X$  = fraction of the desired component

$\theta$  = phase ratio

Solvent extraction can be performed in batch, semi-batch or continuous mode. A continuous solvent extraction process is presented in Figure 3.6. In this process, the material to be extracted is placed in an extraction vessel (i.e. extractor) into which the solvent is introduced at a certain temperature and flow rate. The solvent is then passed into a vessel (i.e. separator) in which the solvent and the extracted compounds are separated, generally by evaporation and finally vacuum distillation. The solvent vapor is then sent to a condenser to be recycled and crude oil is submitted to a refining process (i.e. degumming, alkali refining, bleaching, and deodorization) (Rosenthal et al.,



**Table 3.2** Requirements for solvent extraction of oils

Requirements (per tonne of oilseeds)	Batch processing	Continuous processing
Steam, kg	700	280
Power, kW/h	45	55
Water, m <sup>3</sup>	14	12
Solvent, kg	5	4

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1996). The requirements for batch and continuous extraction mode are presented in Table 3.2.

The efficiency of solvent extraction strongly depends on the solid material condition, the diffusion rate in the solid, the liquid-to-solid ratio, the temperature, the solvent selection (i.e. type, viscosity, and flow rate), the solid's water content, and the presence of competing extractable components (Fellows, 2009; Rydberg et al., 2004; Takeuchi et al., 2008). A pretreatment step like grinding or flaking, prior to extraction, enhances surface contact between the solvent and the solid matrix and thus extraction efficacy. Also, a higher liquid-to-solid ratio provides an increased gradient which facilitates the solute's diffusion. High temperatures increase the solute's solubility and diffusion rate and result in a higher mass transfer rate. Residual water in solid material can negatively affect the solvent's capacity to dissolve solutes and thus, affect the mass transfer. However, the major factor influencing the efficiency of solvent extraction is the nature of the solvent used. For this reason, most extraction techniques manipulate the physical properties of solvents in order to reduce the surface tension, increase the solute's solubility, and promote a higher diffusion rate (Takeuchi et al., 2008).

Solvents range from polar, meaning miscible with water (e.g. ethanol, methanol), to non-polar, which means completely immiscible with water (e.g. hexane). Thus, polar compounds are more soluble in polar solvents whereas non-polar compounds are more readily dissolved in non-polar ones. Selection of the solvent is therefore based on the chemistry of the compound of interest as well as on cost and toxicity. Table 3.3 shows the dielectric constant (DC) of various solvents – a parameter related to polarity or their ability to separate charged molecules (Voet & Voet, 2005). Non-polar solvents (e.g. hexane) have very low DC, while polar solvents (e.g. ethanol, water) have very high DC. For example, water is used to extract sugar, coffee

**Table 3.3** Dielectric constant of solvents used for extraction

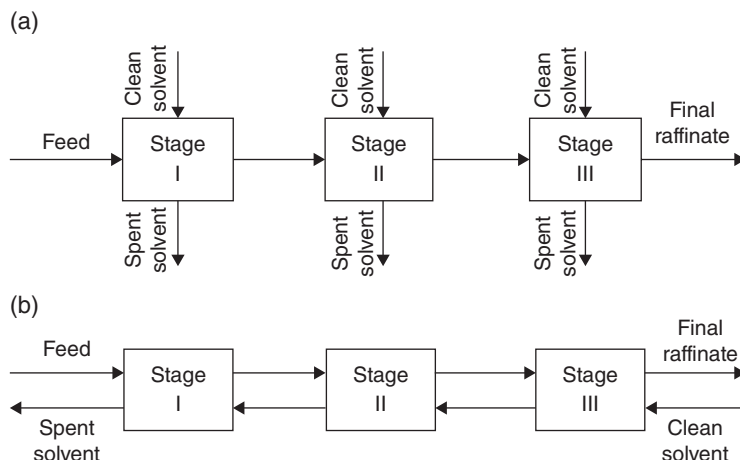
Solvent	Dielectric constant	Type of molecules dissolved
Hexane	1.9	Oils
Toluene	2.4	Oils
Ethanol	24.3	Polyphenols
Methanol	32.6	Polyphenols
Water	78.5	Salts, sugar

Based on Voet and Voet (2005).

and tea solutes, but oil and fat extractions require a less polar solvent, generally hexane.

Another important factor influencing the extraction quality is the number of extraction steps or stages. Indeed, efficiency of the extraction increases along with their numbers. Although single stages have low operating costs, this type of extractor produces diluted solutions involving the use of expensive solvent recovery systems (Fellows, 2009). They are rarely used commercially and are restrained to the extraction of specialty oils or to the production of coffee and tea extracts. Multistage apparatus can be viewed as single-stage extractors linked together, allowing the solvent emerging from an extractor's bases to be pumped cross-currently or counter-currently to the next one (Fellows, 2009). Multistage apparatus offers significant advantages such as higher recovery and purity (Rydberg et al., 2004). In the cross-current mode (Figure 3.7a), the feed, and thereafter the raffinate or residue, are treated in successive stages with fresh solvent. Though operation in cross-current mode offers more flexibility, it is not very desirable due to the high solvent requirements and low extraction yields (Kumar, 2009). For larger volume operations and more efficient uses of solvent, a counter-current mode is employed (Figure 3.7b). In counter-current operation, the feed enters the first stage as the final extract leaves. The last stage receive the fresh solvent as the final raffinate leaves. The counter-current operation provides a higher driving force for solute's mass transfer and thus, gives an optimal performance and is the preferred set-up (Berk, 2009c; Kumar, 2009).

Despite its widespread use in food processing, solvent extraction has considerable drawbacks related to solvent costs, toxicity and reactivity. Also, potential environmental problems are associated with their use, storage, and disposal. For these latter reasons, modern environmentally safe and cost-effective extraction techniques are emerging.



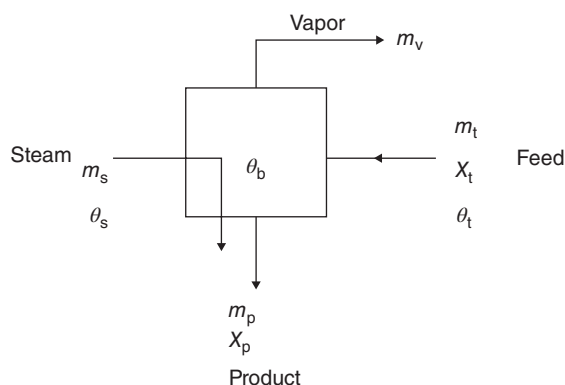
**Figure 3.7** Schematic representation of (a) cross-current and (b) counter-current multiple-stage solvent extraction. Adapted from Li et al. (2012).

Amongst these, supercritical fluid extraction (SFE), pressurized liquid extraction, pressurized hot water extraction (i.e. subcritical water extraction (SWE)), microwave-assisted extraction, and membrane-assisted solvent have been proposed (Lin & Koseoglu, 2005). Even if water is the most environmentally friendly solvent possible, extraction methods using water (e.g. SWE) still give lower yields, require high energy for water removal, and generate large amounts of effluents (Rosenthal et al., 1996).

### 3.3.2 Liquid-gas

#### 3.3.2.1 Evaporation

Evaporation is used in food system to obtain partial separation of a volatile solvent, commonly water, from a non-volatile components. The concentrated liquid obtained possesses an enhanced microbiological and quality stability. It also advantageously reduced storage and transportation costs (Berk, 2009a). This method generally involves the boiling of raw materials and the removal of water vapor. Mass balance (see Equations 3.10–3.11) and heat balance (see Equation 3.12) can be used to calculate the degree of concentration, the operation time, as well as the energy necessary for the process. Indeed, according to the mass balance, “the amount of heat given up by the condensing steam ( $Q$ ) equals the amount of heat used to raise the feed temperature to boiling point and then to boil off the vapor” as expressed by the following equations and as illustrated in Figure 3.8 (Fellows, 2009).



**Figure 3.8** Schematic representation of the mass and heat balance in an evaporator unit (Fellows, 2009).

$$m_f X_f = m_p X_p \quad (3.10)$$

$$m_f = m_p + m_v \quad (3.11)$$

$$Q = m_s \lambda_s \quad (3.12)$$

$$Q = m_f C_p (\theta_b - \theta_f) + m_v \lambda_v$$

Where;

$b$  = food,  $f$  = feed,  $p$  = product,  $s$  = steam and  $v$  = vapor

$C$  = specific heat capacity, J/kg °C

$m$  = mass transfer rate, kg/s

$X$  = solid fraction

$\lambda$  = latent heat, J/kg

$\Theta$  = boiling temperature, °C

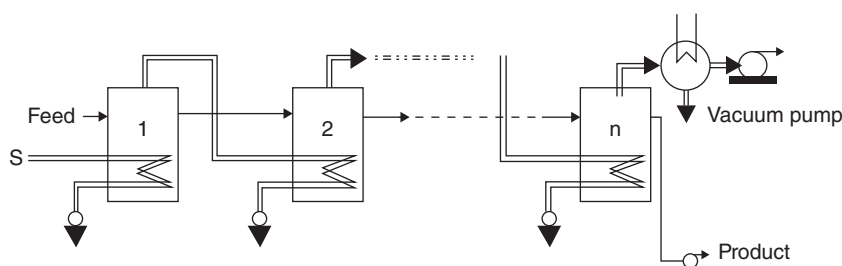


Figure 3.9 Schematic representation of a multiple effect co-current evaporator (Berk, 2009b).

An evaporator is essentially composed of a heat exchanger, or *calandria*, equipped with a device which allows for the separation of vapors from the processed liquid. Heat, commonly from saturated steam, is transferred through a contact surface, generally stainless steel. The driving force for heat transfer is the temperature difference between the steam ( $s$ ) and the feed ( $f$ ). The process is completed when water vapor ( $v$ ) yielded by the product ( $p$ ) is removed as a condensed liquid. The steam generated during evaporation can be reused to heat several other evaporators or stages, in what is called multieffect evaporation (Figure 3.9). This process is especially interesting since it lowers energy costs related to steam production by using the vapor coming from one effect to heat the product in the next effect. However, to be able to perform this in practice, the boiling point of the substance ( $\theta$ ) must be decreased, stage by stage, in order to maintain the temperature difference between the steam and the feed (Barta et al., 2012; Fellows, 2009). The progressive reduction of pressure by the application of a vacuum state increases the heat transfer and allows the product to boil at lower temperatures. Indeed, while evaporation with single-stage devices requires 1.1 kg of steam to evaporate 1 kg of water, only 0.5–0.6 kg of steam is necessary using a two-stage unit (Barta et al., 2012). The application of vacuum evaporation not only reduces energy consumption, it also preserves quality in heat-sensitive products such as milk. In fact, excessive heating time as well as high temperatures can lead to undesired chemical reactions (e.g. Maillard reactions in milk) or to the degradation of compounds (e.g. vitamins).

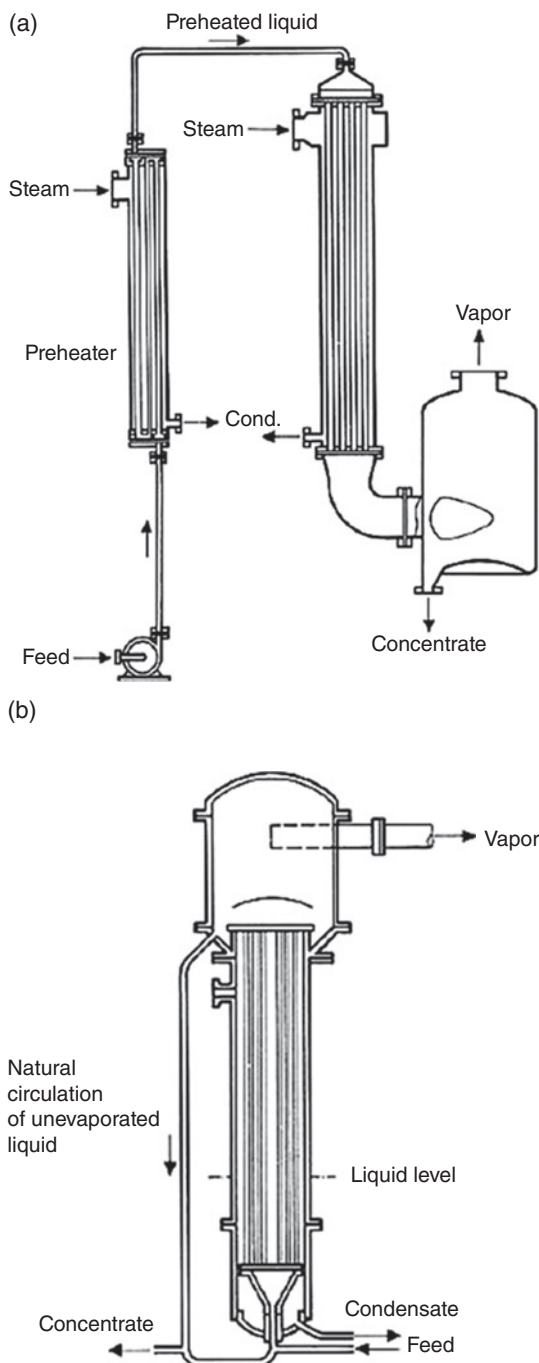
The different types of evaporators used in the food industry include batch pans and boiling film evaporators. Batch pans are the simplest and oldest types of evaporators, consisting essentially of a hemispherical, steam-jacketed vessel. Their heat transfer per unit volume is slower, and they thus require long residence times. Because their heat transfer characteristics are poor—using only natural convection—batch pans have been largely replaced by modern film

evaporation systems. In boiling film evaporators, the product flows as a thin film over a heated surface. Several kinds of film evaporators are available, such as: climbing film, falling film (Figure 3.10), and plate evaporators (Figure 3.11). In climbing film evaporators, the liquid moves rapidly upwards by percolation along vertical tubes and upon reaching the top, the concentrates and vapors produced are sent to a separator. In the case of falling film evaporators, a thin liquid film of uniform thickness moves downwards by gravity inside tubes. Compared to the climbing film evaporator, the residence time of the product is shorter, and this type of apparatus allows a great number of effects (Singh & Heldman, 2008). The same rising and falling principles of evaporation can be used on heat exchange surfaces as a series of plates (i.e. plate evaporators), as shown in Figure 3.11.

A good example of the use of evaporation in the food industry is the manufacture of tomato paste. Tomato paste originates from tomato juice (i.e. 5–6% of solids) in which water is removed by evaporation to reach 35–37% of solids (Singh & Heldman, 2008). Condensed milk and concentrated fruit juices are other examples of food products involving evaporation processes. Compared to membrane processes, a higher degree of concentration is attainable using evaporation (i.e. around 85% compared to 30%), but it involves high energy costs and product quality losses (Kazmi, 2012). Compared to freeze-drying processes, evaporation requires 10–15 times more energy for water removal. However, certain technological problems hamper the widespread application of freeze concentration in the food industry. Indeed, freeze concentration involves expensive systems, considerable loss of solids, and low degree of concentration (i.e. maximum of 50–55% of solids).

### 3.3.2.2 Supercritical fluid extraction (SFE)

Supercritical fluid (SCF) is the state in which the liquid and the gas phases are indistinguishable and in which the compound exhibits properties of both phases.



**Figure 3.10** Schematic representation of two types of evaporator: (a) falling film and (b) climbing film. Adapted from Berk (2009b).

This phenomenon occurs above a critical pressure and temperature, as seen in a phase diagram (Figure 3.12). As expressed mathematically in Equation 3.13, since at low pressure values the densities ( $\rho$ ) and gas are low, the solubility parameter ( $\delta$ ) is also low. As pressure increases, the density of gas grows, reaching the critical point (See Figure 3.12) where the densities of the gas and the liquid are the same (Berk, 2009c). The phase diagram shows that there is a continuous transition from the liquid to the SCF state by increasing the temperature at a constant pressure, or from a gas to the SCF state by increasing pressure at a constant temperature (Turner, 2006). The dependent relation between pressure and the SCF state is the phenomenon on which supercritical extraction is based.

$$\delta = 1.25p_c^{0.5} \left( \frac{\rho_g}{\rho_l} \right) \quad (3.13)$$

Where;

$g$  = gas and  $l$  = liquid

$p_c$  = critical pressure

$\rho$  = solvent density

$\delta$  = solubility parameter

Compared to a liquid solvent, a SCF has a lower viscosity and diffuses easily like a gas. Its lower surface tension allows for rapid penetration of the food material and thus, increases extraction efficiency. Furthermore, SCF has a higher volatility which allows its complete separation, avoiding the presence of residual solvent in the food material. Favorably, like a liquid, the SCF can dissolve large quantities of desired molecules, and thus extract them effectively (Berk, 2009c).

Supercritical fluid extraction (SFE) is an extraction process carried out using a supercritical fluid as a solvent (Berk, 2009c). In the food industry, carbon dioxide ( $\text{CO}_2$ ) is the most common SCF used for the extraction of compounds. SFE represents an eco-friendly and cost-effective alternative to a chemical solvent, since it is non-toxic, can be used without declaration, is non-flammable, inexpensive, readily available, environmentally acceptable, chemically inert and liquefiable under reasonable pressure (Brunner, 2005). Indeed,  $\text{CO}_2$  reaches its SCF state beyond a critical point at a temperature of  $31.1^\circ\text{C}$  and a pressure of 73.8 bar, as shown in Figure 3.12 (Fellows, 2009). Below this critical point, liquid  $\text{CO}_2$  (e.g. subcritical state) behaves like any other liquid, while above this critical point, it only exists in the SCF state and acts as a gas.

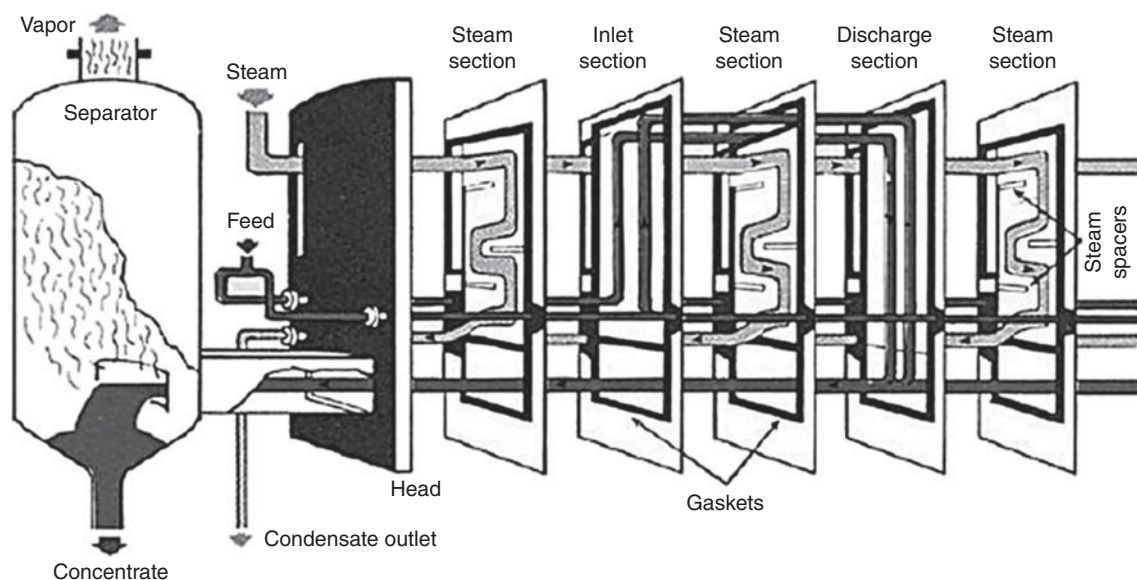


Figure 3.11 Schematic representation of a plate evaporator (Berk, 2009b).

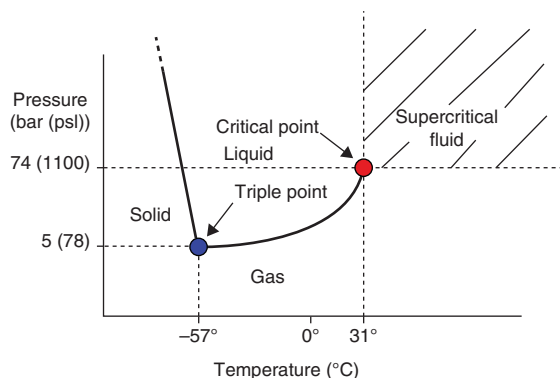


Figure 3.12 Pressure-temperature phase diagram for CO<sub>2</sub>. Adapted from Fellows (2009).

The SFE apparatus is similar to the one used for solvent extraction (see section 3.3.1.3). However, the extraction and separation vessels of SFE apparatus are pressurized chambers equipped with heat exchangers since the state of CO<sub>2</sub> is determined by pressure and temperature. The CO<sub>2</sub> is stored in a subcritical state (i.e. liquid state) inside the condenser and pumped into the extraction vessel throughout a heat exchanger by a high-pressure pump. In the extraction vessel, as a result of higher pressure

and temperature, CO<sub>2</sub> reaches the SCF state before being mixed in the food material (Fellows, 2009). Afterwards, SCF-CO<sub>2</sub> passes into the separation vessel where the pressure is lowered, allowing for the return of CO<sub>2</sub> to its gaseous state and thus for the precipitation of the dissolved solutes in the separation vessel (see Figure 3.12). Finally, the extracted compounds are removed from the separation vessel as the gaseous CO<sub>2</sub> is sent to a condenser to be recycled and stored in its subcritical state (e.g. liquid state) by lowering the temperature.

Supercritical fluid extraction can nowadays be performed at an analytical, pilot scale as well as in large-scale industrial plants. More than 100 industrial plants and around 500 pilot plants are using this technology worldwide (Turner, 2006). Supercritical processes can be used to extract a wide variety of molecules such as lipids (e.g. seed oil, fish oil, specific fractions of butter fat or specific essential fatty acids), caffeine from coffee beans and tea leaves (decaffeinated), alcohol from beer and wines, lecithin, bioactive compounds (i.e. antioxidants, phytosterol, and vitamins) and various flavors, colorants, and fragrances (Sahena et al., 2009). This extraction technology offers extraction yields comparable to those of conventional solvent extractions, and can be carried out in different modes of operation (e.g. batch, single stage, multistage, usually in counter-current mode).

In the food industry, large-scale uses are mainly for the decaffeination of coffee beans and black tea as well as for the removal of bitter flavors from hops (Rizvi, 2010). For example, Kaffee HAG AG, originally from Bremen, Germany, is a worldwide brand of SFE-CO<sub>2</sub> decaffeinated coffee owned by US multinational Kraft Foods (50,000 ton/year of coffee) (Otles, 2008). However, extractions of added-value components such as specific fatty acids and essential oils are still limited to smaller scale plants (Brunner, 2005). Overall, even if SFE using CO<sub>2</sub> is advantageous in terms of safety (i.e. food quality and environment) and operating costs, the industrial use of this technology is limited due to high investment costs.

### 3.4 Membrane separations

#### 3.4.1 Pressure-driven processes

##### 3.4.1.1 Basic principles and separation ranges (RO, NF, UF, MF)

Pressure-driven membrane separation processes have been integrated as unit operations into a large number of food processes and it is one of the fastest growing technologies in the field of separation methods. Membrane technology requires low capital as well as low utility costs, and for this reason membrane separation has replaced the conventional separation technique in many food processes. In fact, conventional separation and concentration techniques usually imply energy-consuming phase changes which can affect both the physical and chemical characteristics of the final product. Furthermore, membrane systems, when compared with the conventional technology of separation and concentration, only require

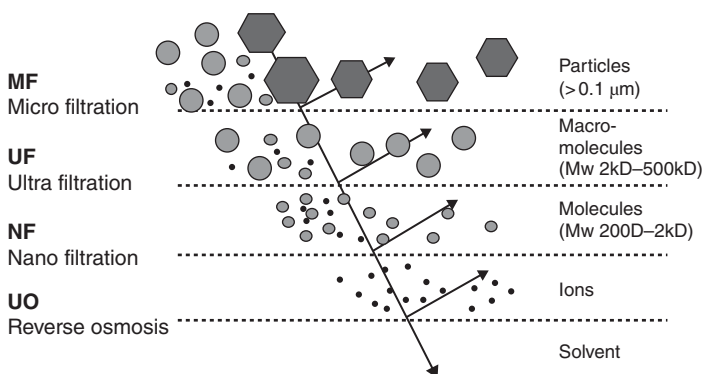
limited space and thus do not involve expensive installations (Philipina & Syed, 2008).

Membrane processes are classified based on four main ranges of separation, namely: microfiltration (MF) (0.1–5 µm, 1–10 bar), ultrafiltration (UF) (1–100 nm, 1–10 bar), nanofiltration (NF) (0.5–10 nm, 10–30 bar) and reverse osmosis (RO) (<0.5 nm, 35–100 bar) (Cui et al., 2010) as illustrated in Figure 3.13. Pressure-driven membrane separation operates at a pressure that varies inversely with pore size.

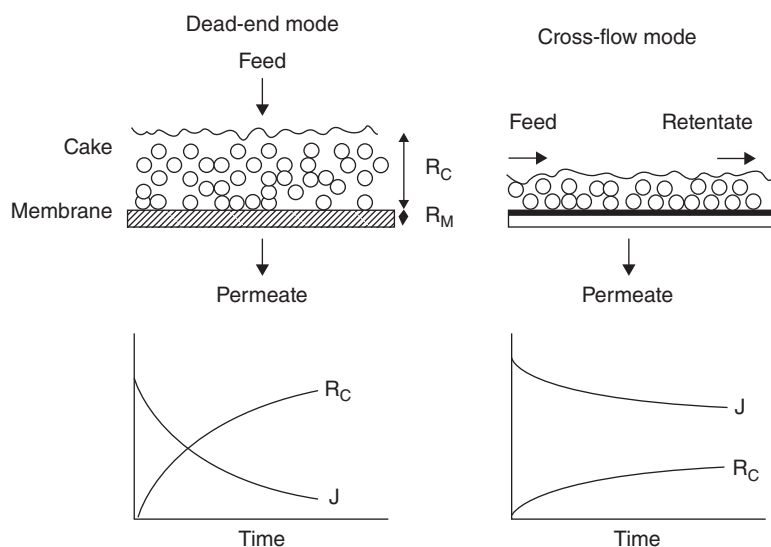
Pressure-driven membrane separation can be performed in dead-end mode or in tangential flow or cross-flow (Figure 3.14). Since in dead-end mode all the fluids to be filtered pass through the membrane's surface, trapped particles can build a filter cake and thus reduce filtration efficiency. For this reason, industrial-scale installations operate in the tangential flow mode in which the solution circulates tangential to the membrane's surface. In tangential mode, the particles rejected by the membrane (i.e. retentate) continue to flow, preventing the formation of thick filter cakes and thus, helping to maintain a more constant flux ( $J$ ) of fluid passing through the membrane (i.e. permeate) and a more steady retention factor ( $R$ ).

##### 3.4.1.2 Membrane configurations, operation modes, process design

The availability and characteristics of membrane materials and filtration modules are constantly evolving, making it challenging to select a suitable membrane for a given application. Table 3.4 lists the main characteristics of commercially available membranes. A membrane material can be characterized by either its hydrophobicity, which minimizes fouling, or by its strength and durability



**Figure 3.13** Separation range of tangential flow membrane processes.



**Figure 3.14** Schematic diagram of dead-end and cross-flow filtration modes and their impact on permeation flux ( $J$ ) and cake formation (Cui et al., 2010).

**Table 3.4** Characteristics of commercially available membranes

Material	Range				Resistance			Configuration*			
	MF	UF	NF	RO	Temp	pH	Chlorine**	Sp	Hf	T	Pl
Polysulfone	✓	✓	✓		80	0–14	M	✓	✓		✓
Polyamide	✓	✓	✓	✓	80	0–14	M	✓			✓
Cellulose acetate	✓	✓	✓	✓	80	2–8	L	✓	✓		✓
Ceramic	✓	✓	✓		1000	0–14	H			✓	
Carbon	✓	✓	✓		1000	0–14	H			✓	

\*Sp, spiral; Hf, hollow fiber; T, tubular; Pl, planar module.

\*\*L = low; M = medium; H = high.

regarding mechanical breakdowns and intensive cleaning. No material satisfies both characteristics, but polyvinylidene fluoride (PVDF) membranes have gained in popularity for their long life span (i.e. 3–5 years for conventional applications and 5–10 years for water clarification uses) (Kubota et al., 2008). Most membrane materials are available for all four ranges of separation.

Membrane modules are commercially available in four configurations: planar or flat sheet, tubular, hollow fiber, and spiral-wound modules (Sinnott, 2005). The choice of configuration is based on economic considerations associated with performance (i.e. pressure drop, resistance to fouling), surface/volume ratio, cost of cartridge replacement, and ease of cleaning and replacement (Fellows, 2009). Spiral membranes are preferred

for their low cost and high surface/volume ratio compared to tubular elements. However, they can easily be blocked by suspended particles, and thus require relatively clean feeds (Sinnott, 2005). Tubular modules have the lowest surface/volume ratio but their large internal diameter allows for the treatment of feeds containing large particles. Flat sheet modules permit the stacking of several membrane units, although the resulting pressure drop can compromise the process performance. This type of module lies between tubular and spiral-wound modules in term of cost and energy consumption. Despite the fact that hollow-fiber membranes are easy to clean, have the highest surface/volume ratio, and the lowest energy cost among all membrane configurations, this type of module has the disadvantage of confining liquid flow



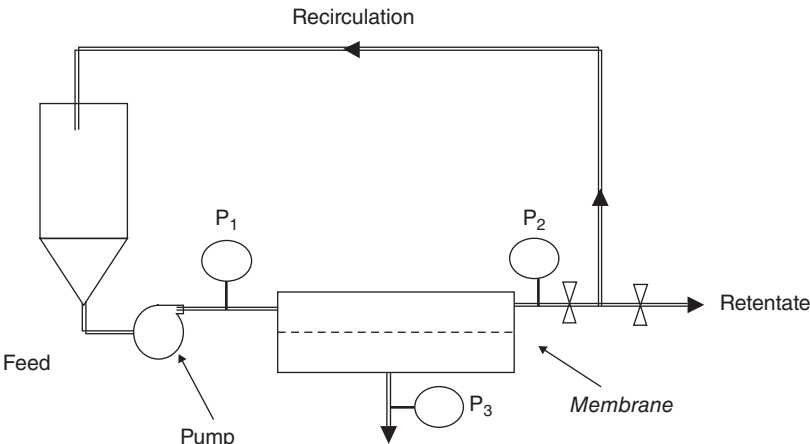


Figure 3.15 Schematic diagram of a simplified filtration system.

Table 3.5 Typical performance parameters of pressure-driven membrane processes

	Membrane type			
	MF	UF	NF	RO
Pore size	0.1–5 $\mu\text{m}$	1–100 nm	0.5–10 nm	<0.5 nm
Smallest particles removed	Colloids, bacteria	Large organic molecules, viruses	Small organic molecules, divalent ions	All dissolved species
Operating pressure (bar)	1–10	1–10	10–30	35–100
Permeation flux ( $\text{L}/\text{m}^2\cdot\text{h}$ )	100–1000	50–200	20–50	10–50

Based on Cui et al. (2010) and Jirjis & Luque (2010).  
MF, microfiltration; NF, nanofiltration; RO, reverse osmosis; UF, ultrafiltration.

through narrow veins (i.e. inner diameter <1–2 mm) – limiting their use to liquids free from visible suspended particles. For this reason, hollow-fiber membranes are mainly used for RO applications such as desalination (Fellows, 2009) or require a pretreatment to reduce particle sizes to 100  $\mu\text{m}$  (Sinnott, 2005).

As depicted in Figure 3.15, most installations for membrane-based separation processes include the following: (1) a feed tank, (2) the membrane, (3) at least one pump, and (4) two manometers located at the inlet ( $P_1$ ) and outlet ( $P_2$ ) of the membrane compartment. The transmembrane pressure ( $TMP$ ) constitutes the driving force of the filtration and indicates the pressure drop associated with permeation. The permeation flux ( $J$ ) provides an estimation of the overall performance of the filtration system by indicating the rate of mass transport across the membrane (Cui et al., 2010).

As expressed mathematically in Equation 3.14, the permeation flux allows for comparison of data from different membrane systems (see Table 3.5). The recirculation speed ( $v$ ) also constitutes a critical parameter of operation, since it can be adjusted to maintain a turbulent flow regime and thereby maximize membrane surface sweeping and slow down membrane fouling.

The separation capacity of UF and NF membranes is determined mainly by their molecular weight cut-off (MWCO). MWCO indicates the molecular weight (Da) of the species rejected in a proportion of 90–95% (Takeuchi et al., 2008). Even though the MWCO and pore diameter constitute reference values for membrane selection, it remains essential to determine the rejection coefficient or retention factor ( $R$ ) for the molecular species or solutes being concentrated



(Equation 3.15) (Cui et al., 2010). For example, a retention factor of 1.00 indicates that the membrane rejects 100% of the solute molecules, while a value of 0 indicates that the membrane is totally permeable to them. RO membranes are often graded using salt (e.g. NaCl or CaCl<sub>2</sub>) passage data. Finally, the volume concentration factor (VCF) represents the ratio of the initial solution volume ( $V_0$ ) and the final volume of the concentrate obtained ( $V_r$ ) as expressed in Equation 3.17 (Cui et al., 2010). For example, a VCF value of 3 indicates that 90 L of a solution was concentrated to a final volume of 30 L. In addition, it is useful to estimate the concentration factor (CF) of a product (i.e. the final product concentration/initial product concentration) as a function of VCF for a given species of which the rejection coefficient ( $R$ ) is known (see Equation 3.18).

$$J = \frac{\Delta V}{\Delta t \times A} \quad (3.14)$$

$$R = 1 - \left( \frac{C_p}{C_r} \right) \quad (3.15)$$

$$TMP = \frac{(p_r - p_p)_{in} - (p_r - p_p)_{out}}{2} \quad (3.16)$$

$$VCF = \frac{V_0}{V_r} \quad (3.17)$$

$$CF = (VCF)^R \quad (3.18)$$

Where;

$p$  = permeate and  $r$  = retentate

$A$  = membrane area, m<sup>2</sup>

$C$  = concentration

$CF$  = concentration factor

$J$  = flux, L/m<sup>2</sup>h

$R$  = retention factor

$t$  = time, h

$TMP$  = transmembrane pressure

$V$  = volume, L

$VCF$  = volume concentration factor

Membrane separations can be operated in batch or continuous mode. Comparison between the two modes must be made according to several criteria associated with cost and productivity constraints, but also with processing time. In fact, the latter must be minimized to prevent excessive bacterial growth, oxidation of fat, and denaturation of protein constituents due to mechanical shear stress resulting from recirculation.

Figure 3.16 shows a comparison of the batch (a) and continuous modes (b). Batch concentration is schematized

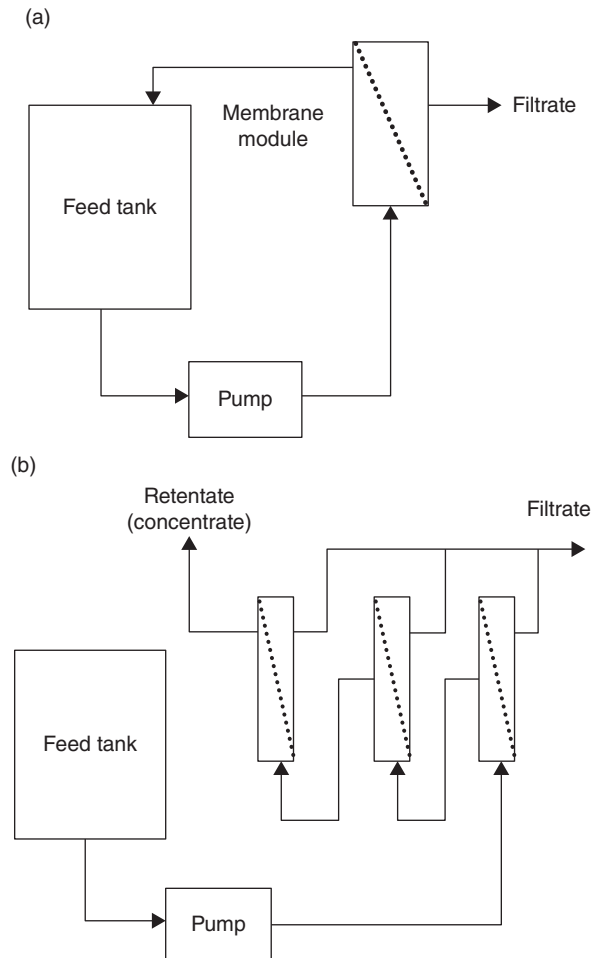
by a closed loop in which the retentate is returned to the feed tank and the permeate is removed continuously until the desired VCF is reached. A retentate recirculation loop can be inserted to increase the tangential speed of the fluid and thus maintain a higher mean flux. This also decreases the power required from the feeding pump and decreases general operating costs. However, for high concentration retentate using a recirculation loop, long residence time can lead to microbiological problems since temperature used is often around 50°C. For this reason, batch mode used is generally well adapted for small-scale applications (Jirjis & Luque, 2010).

The continuous process is characterized by feeding the solution to be treated at the same rate as the concentrate removed in what is termed as: *feed and bleed*. This operation mode offers the main advantages of faster processing, retentate of more uniform quality, continuous production of the final product, and lower feeding tank capacity. In addition, it offers the possibility of juxtaposing multistage filtration loops in which retentate from a stage feeds the next one. Using membranes of different molecular weight cut-off at each stage, the multistage approach allows continuous concentration and purification of molecules of different molecular weights.

#### 3.4.1.3 Polarization and fouling phenomena

With processing time, performance of filtration operations will inevitably decrease as an extra resistance adds up to the membrane resistance. Two phenomena can cause a decrease in the permeation flux ( $J$ ) during filtration processes: (1) polarization and (2) fouling (Li & Chen, 2010), as represented in Figure 3.17.

Although there is no universal definition, polarization can be described as the reversible accumulation of dissolved or suspended species near the membrane's surface (Li & Chen, 2010). This phenomenon can usually be controlled by adjusting hydraulic parameters (i.e. pressure, speed of recirculation) to re-establish the permeate flux ( $J$ ). Fouling refers as the irreversible formation of a deposit of retained particles in the membrane pores (i.e. pore blocking) or surface (i.e. absorption). It results in unstable filtration behaviors. In this case, the permeate flux ( $J$ ) can only be re-established by interrupting the process for membrane cleaning. Since membrane cleaning involves operation as well as energy costs and affects the lifetime of membranes fouling is often the main limitation of membrane applications in the food industries (Li & Chen, 2010). Fouling can be caused by



**Figure 3.16** Schematic diagrams of (a) batch and (b) continuous filtration operation modes (Raja, 2008).

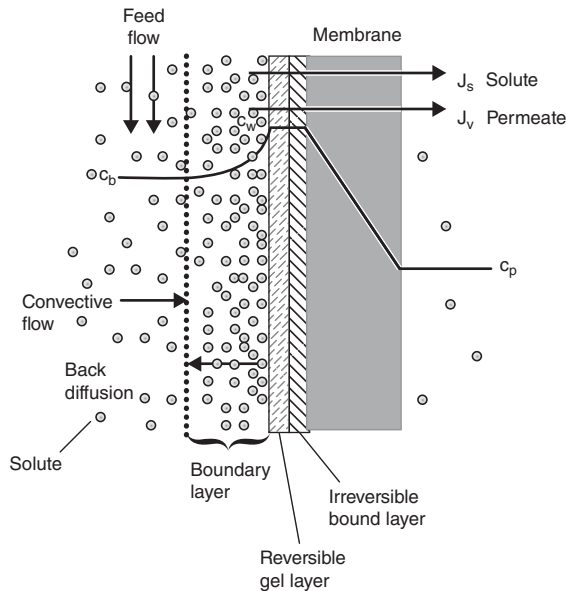
inorganic compounds (e.g. minerals), microorganisms (e.g. biofilms) or macromolecules (e.g. proteins, carbohydrates, and fats). This undesired phenomenon affecting productivity, as well as membrane selectivity, can however be controlled by several parameters such as: the nature and concentration of the feed, the type of membrane used, the pore size distribution, membrane material, and operational conditions (e.g. filtration mode, transmembrane pressure (TMP), temperature, turbulence, etc.) (Cui et al., 2010; Li & Chen, 2010). Figure 3.18 illustrates the effect of TMP, recirculation speed ( $v$ ), protein concentration, and temperature on permeation flux ( $J$ ) in the ultrafiltration of sweet whey.

Amongst all factors influencing fouling phenomena, three major groups are recognized: (1) nature and

concentration of the feed; (2) type of membrane and membrane material; and (3) operational conditions.

#### 3.4.1.3.1 Nature and concentration of the feed

Fouling phenomena are generally increased in high concentration feed, as the amount of proteins decrease permeation flux ( $J$ ) via an increase in solution viscosity, but mainly by the accumulation of protein in the polarization concentration zone (see Figure 3.17). Indeed, macromolecules (e.g. proteins) can form a gel layer at high concentration or cause cake formation and pore blocking, as hydrophobic molecules (e.g. fatty acids) can be absorbed by the membrane surface. However,



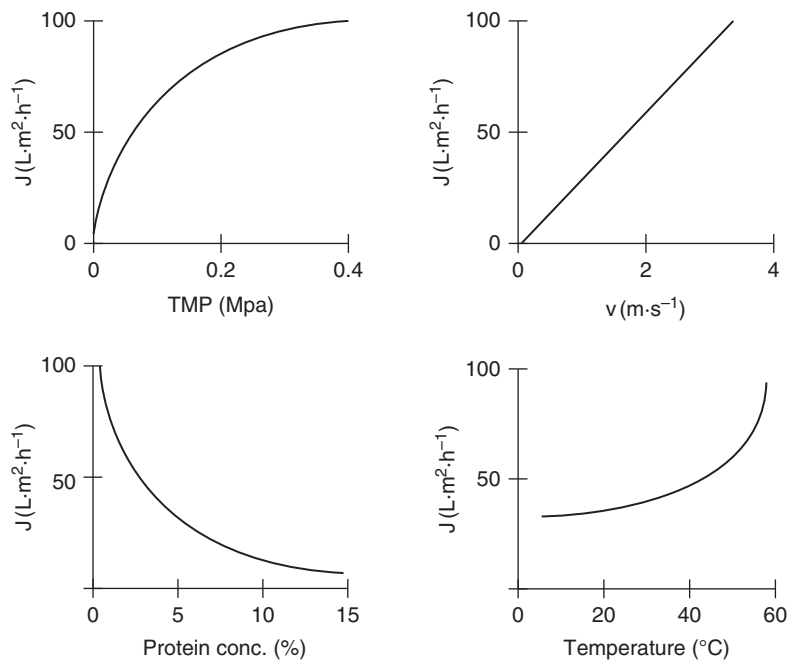
**Figure 3.17** Schematic representation of polarization and fouling phenomena (Goosen et al., 2005).

depending of the nature of the feed, high concentration can, in some cases, reduce fouling tendency by promoting molecule aggregation.

It is possible to minimize fouling by pretreating the feed by using operations such as prefiltration, heat treatment, chemical clarification, chlorination, pH adjustment or addition of complexing agents (Beltsios et al., 2008). Those pretreatments affect the structure, ionic conditions, charges and concentration of molecules present in the feed and thus affect the tendency towards fouling. The following examples apply to milk and whey processing, but similar principles of pretreatment can be applied to the filtration of other foodstuffs.

- **Prefiltration/centrifugation:** although the majority of filtration systems include prefilters, fluids such as whey may contain suspended particles (i.e. casein fines) and residual lipids that slip through and foul membranes. Treatment of whey using a conventional clarifier helps to prevent this phenomenon.

- **pH adjustment:** decreasing the pH of dairy fluids between 5.5 and 6.5 can increase permeation flux significantly. Indeed, this keeps calcium phosphate in solution and thus, prevents the collection of precipitate in the membrane structure. It should be noted that decreasing



**Figure 3.18** Effect of operating conditions on the permeation flux in UF (example of sweet whey). Adapted from Walstra (1999).

pH usually increases the concentration of free calcium ions ( $\text{Ca}^{2+}$ ), which can promote fouling, especially in the case of ceramic membranes.

- **Preheating:** the positive effects of preheating (50–60°C for 30–120 min) on permeation flux are widely exploited in the manufacture of whey protein concentrates by UF. Heating causes excess calcium phosphate to precipitate, the free calcium ion concentration to decrease, and lipoproteins to aggregate. Clarification is often needed to remove precipitated material from preheated whey.
- **Defatting:** defatting consists of decreasing the concentration of residual lipids, for example in a dairy fluid. It is typically a combination of pH adjustments, heating and clarification or centrifugation. It is possible to obtain defatted dairy fluids (including milk and buttermilk) by tangential microfiltration, using a membrane of 1.4  $\mu\text{m}$  pore size.
- **Demineralization/calcium ion sequestration:** demineralization is also an effective means of preventing the precipitation of calcium phosphate. Chemicals that sequester calcium (e.g. EDTA, citric acid) can be used for this purpose, but it must be established first that the sequestering agent has no affinity with the membrane material.

#### 3.4.1.3.2 Type of membrane and membrane material

Membrane properties such as materials, surface morphological structures (e.g. heterogeneity of the pores and pore sizes) and surface properties (e.g. smoothness of the surfaces, hydrophobicity or surface charges) can all modulate the tendency toward fouling (Brunner, 2005). For example, hydrophobic membranes (e.g. polysulfone) will tend to adsorb proteins, as hydrophilic ones (e.g. cellulose acetate) will have a higher affinity for minerals (e.g. calcium) and be less inclined to fouling. For the reason, a hydrophilic coat is frequently applied on hydrophobic membranes in order to minimize fouling phenomena (Li & Chen, 2010).

#### 3.4.1.3.3 Operational conditions

The type of filtration mode and the optimization of operational conditions can also minimize fouling. For example, compared to cross-flow filtration – in which the feed runs tangential to the membrane – dead-end filtration will promote cake formation, as large molecules (i.e. larger than the pores) are stopped at the membrane's surface. Indeed, cross-flow technique prevents filter cake formation (see Figure 3.14).

Adjustments of TMP, temperature of operation as well as the use of turbulence promoters can all decrease the tendency toward fouling. An increased pressure can compact the existing filter cake and thus, negatively affect the fouling phenomena. An higher recirculation speed amplifies the shear rate near the membrane's surface and reduces the risk of protein gelling. Similarly, an increased in TMP can sometimes optimize the permeate flux in cases where recirculation speed is sufficient to maintain a turbulent regime. However, in some case, but in some cases, the resulting increases of the driving force favorably affect polarization by increasing the foulant's compaction (Li & Chen, 2010). An higher temperature can also lead to an improvement in permeate flux by reducing viscosity and increasing the permeability of the membrane material. However, it is important to remember that processing temperatures above 60°C denature many proteins, which not only affects their functional properties but will also make them more liable to participate in irreversible fouling of the membrane. Moreover, the temperature is limited by the thermal resistance of the membrane material (Li & Chen, 2010). Finally, it is possible to reduce membrane fouling with: (1) the use of turbulence promoters, which decreases concentration near the surface, and (2) backflushing, which removes cake layers (Cui et al., 2010). An electric field across the membrane (i.e. electrofiltration), pulsed flow (i.e. fluctuating pressure), and rotating or vibrating dynamic membrane systems are all used to reduce fouling and the polarization phenomena (Fane & Chang, 2008).

In conclusion, control of fouling and polarization phenomena can be achieved by controlling the composition and physicochemical properties of the fluid to be processed and by optimizing the filtration system's performance parameters.

#### 3.4.1.4 Applications of membranes in food processing

Membranes used by the food processing industry represent 20–30% of all worldwide membrane sales. This market is growing at a fast annual rate of around 7.5% (Mohammad et al., 2012). Mainly used in the dairy industry (close to 40% of all use), membrane technology is also used for beverages, sugar refining, and oil processing. Membrane processes are advantageous for environmental, competitive and economic reasons, and they also allow for the production of high-quality food products, from a nutritional and food safety point of view. They are mainly used for concentration purposes in the food industry. Around 58% of the membrane market is represented by MF membrane, followed by UF and RO membranes (around 17% each).

The remaining 8% represents the other membrane technologies, such as NF, pervaporation (2%), and electrodialysis. Moreover, membrane technologies simplify conventional processing methods by removing processing steps, are easy to operate, and can improve process performances (e.g. clarification) (Daufin et al., 2001). RO represents the most economical preconcentration method in food processing and thus can generate enormous energy savings for industries (Munir, 2006).

#### 3.4.1.4.1 Dairy industry (UF or MF of milk, cheese whey, dairy effluents)

Milk is a complex mixture of different types of molecules including proteins, lipids, lactose and minerals, but also contains undesirable components such as bacteria. In the dairy industry, membrane technologies are used to concentrate and separate milk and milk by-product components, thus adding to their commercial value. Actually, whey processing represents the main application of membrane technology in the dairy industry, with more than 75% of all membrane usages (Mohammad et al., 2012).

Tangential flow or cross-flow MF, the uniform transmembrane pressure (UTP) concept, and ceramic-based membrane improvements have all contributed to the increased utilization of MF in dairy processing (Saboya & Maubois, 2000). There are three major applications of MF in the dairy industry: (1) the removal of bacteria from milk; (2) the pretreatment of cheese whey (i.e. defatting and removal of bacteria); and (3) the micellar casein enrichment of cheese milk. Indeed, in the dairy industries, MF is used to produce longer shelf life products (i.e. 16–21 days compared to 6–8 days for conventional pasteurization processes) without cooked off-tastes and with a greater reduction of spore-forming bacteria (Philipina & Syed, 2008). In fact, 99.99% of bacteria can be removed from skim milk using a commercial MF process called Bactocatch® (Tetra Laval, Lund, Sweden). This process, employing an 1.4 µm MF membrane (50 °C, 0.5 bar), has been used commercially mainly in Canada and western Europe (Elwell & Barbano, 2006). It should be noted that, for legal as well as safety considerations, all MF milk commercialized in U.S. and Canada needs to be further pasteurized.

Microfiltration has also been proposed as an effective method to remove fat from whey before further processing by UF. Indeed, compared to the conventional pretreatments used in the industry (i.e. clarification followed by pasteurization), MF removes phospholipoprotein complexes as well as all residual lipids, even

the smallest milk fat globules (Li & Chen, 2010). It is possible to increase the UF permeation flux by 30% by pretreating cheese whey using MF before concentration. Moreover, MF can retain valuable whey proteins such as bovine serum albumin and immunoglobulin-G, which can be useful for functional food applications (Morr & Ha, 1993). The pretreatment of cheese whey by MF allows the production of high-quality, low-fat “undena-tured” whey protein concentrate (WPC), and whey protein isolate (WPI) (Smithers, 2008). Finally, MF can be utilized in cheese making to change the casein-to-whey protein ratio or the casein-to-fat ratio. In fact, the application of the MF process to whole milk allows for the production of an enriched native and micellar calcium phosphocaseinate retentate which improves rennet coagulation and thus, productivity of cheese manufactures (Li & Chen, 2010). Moreover, by using a 3% phosphocaseinate solution, it is possible to reduce the coagulation time by 53% and increase the firmness (after 30 min) of more than 50% in comparison to raw milk (Daufin et al., 2001).

Ultrafiltration is the most commonly used membrane process in the world’s dairy industry (Daufin et al., 2001). The fractionation and concentration of whey protein represent the most important industrial application of UF in the dairy industry. The whey protein UF concentrates can be further purified using DF to obtain high-purity WPC and WPI. Since all proteins in skim are retained and concentrated by UF, concentration and purification of milk protein to produce milk protein concentrates (MPC) and isolates (MPI) also represent a main application of UF in dairy processing. UF-MPC are extensively used by soft cheeses manufacturers as pre-cheese to be coagulated and fermented (Philipina & Syed, 2008). This process allows the full retention of whey proteins in the cheese matrix and thus eliminates whey drainage and reduces rennet requirement by around 80% compared to conventional cheese-making methods (Elwell & Barbano, 2006). However, bitterness problems have been reported in soft cheeses as well as texture defaults in semi-hard and hard cheeses (Elwell & Barbano, 2006). UF of milk generates, as a co-product, protein-free permeate which may subsequently be NF-processed to recover lactose. Finally, UF is also used to standardize milk by the adjustment of the mass ratio to the different milk constituents.

Nanofiltration is somewhat similar to UF and is commonly used in the industry for the processing of UF and MF permeates (Philipina & Syed, 2008). Indeed, the main application of NF in the dairy industry is the desalting

of whey permeates (Munir, 2006). As with RO, it can be used for the preconcentration of milk or whey (by the removal of water and minerals) mainly to reduce transportation costs or energy requirements before the evaporation process (Munir, 2006).

#### 3.4.1.4.2 *Fruit and vegetable juices (clarification and concentration of fruit juice)*

Clarification, concentration, and deacidification are the main uses for membrane technology in fruit juices processing (i.e. around 20% of all membrane usages). Concentration of fruit and vegetable juices has economic advantages for packaging, storage, and distribution. Moreover, membrane processes avoid color degradation and cooked off-tastes, as well as the loss of delicate aromas important to fresh juice flavors (Munir, 2006).

Ultrafiltration is used in the processing of multiple fruit and vegetable juices such as orange, lemon, grapefruit, tangerine, tomato, cucumber, carrot, and mushroom (Mohammad et al., 2012). Typically, juice is extracted using a press and passed through a UF module prior to concentration by evaporation or further membrane processes. UF membranes retain the concentrated pulp fraction (i.e. retentate) as well as unwanted enzymes. The UF-clarified permeate obtained can be pasteurized and, if needed, further concentrated. Most of the bioactive compounds are recovered in the permeate using this technology. Thereafter, pasteurized pulp can be reincorporated to the clarified permeate fraction to obtain whole juice. In the industry, pretreatment of fruit by enzymes (e.g. pectinases and cellulases) is commonly applied to improve fruit juice extraction, to reduce juice viscosity, and improve juice yield and color. The use of immobilized pectinases on UF membrane has been proposed as a method to allow the reuse of enzymes while controlling membrane fouling during clarification processes (Giorno & Drioli, 2000).

Reverse osmosis favorably concentrates fruit juices, giving high-quality products in which both nutritional and sensorial qualities are maintained by the low temperature used in the process. However, RO gives a low concentration level (25°Brix) compared to evaporation (42–65°Brix) (Jesus et al., 2007). Recently, a new method called “high-concentration RO” has been described for the concentration of orange and apple juices, using the combination of two membranes (Echavarría et al., 2011; Munir, 2006). A first membrane retains sugars and aroma components (i.e. retentate), which can then be processed using a second membrane that allows some of the sugars

to pass through. The result is a lower transmembrane osmotic pressure differential and a concentrated retentate (42–60°Brix) with organoleptic properties close to those of fresh juice with no loss of acids, vitamin C, limonene, or pectin (Munir, 2006). This process has some disadvantages including generation of a diluted, low-value by-product, and the need for complex and high-cost systems (Merry, 2010).

#### 3.4.1.4.3 *Sugar refining (concentration, clarification, and purification)*

Traditionally, sugar syrup concentration is performed using evaporation. This is energy consuming and can lower the quality of the sugar and negatively influence its color. Membrane processes represent an alternative to evaporation in sugar refining for concentration purposes. It can also be used for clarification and purification applications. Indeed, raw juices from sugar cane or sugar beet contain not only sucrose, but various polysaccharides, proteins, gums, and other unwanted components. Those impurities are traditionally removed using anionic resins which generate polluting elutes (Daufin et al., 2001). Industrial clarification of raw sugar juices by UF or MF is rapidly growing, due to their greater capacity to remove unwanted macromolecules and microorganisms compared to traditional methods. Membrane processes remove unwanted materials (i.e. retentate) and give a decolorized raw sugar juice (i.e. permeate) ready for concentration and crystallization. Concentration of sugar juices by RO or NF is cost-effective compared to evaporation. The use of NF as a preconcentration step can reduce the loss of sugars in the molasses by 10% (Madaeni et al., 2004; Munir, 2006). RO has been used for the concentration of maple syrup, resulting in more than 30% reduction in processing costs (Munir, 2006).

#### 3.4.1.4.4 *Vegetable oils processing*

The production of edible oils follows a series of steps necessary for proper product quality by the removal of impurities such as water, dust, phospholipids, free fatty acids, gums, waxes, oxidation products, pigments, and trace elements (e.g. iron, copper, and sulfur). These processes include degumming, deacidification/neutralization, bleaching, dewaxing and deodorization. Those latter processing steps mostly involve high temperatures, the use of harsh chemicals and of considerable amounts of energy in the form of steam or electricity. Membrane-based processes (i.e. MF, UF, NF) can

practically replace all the steps necessary for edible oils processing in a simple, competitive and eco-friendly way (Ladhe & Krishna Kumar, 2010). However, development of membranes allowing higher flux and selectivity combined to less fouling is still necessary to the replacement of critical energy-costing steps (i.e. degumming, refining, and bleaching) in commercial applications (Lin & Koseoglu, 2005).

Furthermore, membrane-based technologies can advantageously remove oxidation products as well as heavy metal traces. Indeed, with regard to their ability to remove oxidation products, membrane processes have been proposed as a method to extend the life-span of frying oils by the removal of proteins, carbohydrates and their decomposition products, and prevent color changes (Snape & Nakajima, 1996).

Membrane technologies can also be used to recover vegetable protein from oil extracting residues. For example, UF has been successfully applied to obtain soy protein isolates (i.e. 60–65% of proteins) from the defatted soybean meal or “cake” (Ladhe & Krishna Kumar, 2010). The isolate obtained presents good nutritional value and can be used in breakfast cereals, animal nutrition, confectionery, and dairy imitation products, as well as in nutritional and dietary beverages (Koseoglu & Engelgau, 1990).

Although membrane technologies can advantageously replace some of the steps in edible oils processing, only a few commercial applications have been reported. Nitrogen production for packaging uses, waste water treatment using UF/RO and phospholipids removal using MF have been described in literature (Lin & Koseoglu, 2005).

#### 3.4.1.4.5 *Brewing and wine industry*

Beer is the second most consumed beverage in the world. This industry is constantly being challenged to create products with consistent quality and unique taste while maintaining low production costs and environmentally friendly process (Carmen & Ernst Ulrich, 2008). Membrane technology applications are emerging as they enhance product quality, are energy saving, and reduce water waste.

The production of beer involves mashing, boiling, fermentation, and maturation steps followed by a pasteurized process to ensure microbiological stability and conservation. During brewing, several filtration operations are essential to remove solids particles (e.g. yeasts, malt, and hops) from the product. This separation of solid from

liquid, traditionally done by dead-end filtration on diatomaceous earth (DE), represents a challenge for economic, environmental, and technical purposes. Indeed, the DE used for traditional filtration is difficult to handle and thus represents a potential health hazard. Also, it needs to be properly disposed after usage and involves additional filtration steps.

Microfiltration is mainly used in the brewing industry for the recovery of beer from fermentation and maturation tank bottoms, and is considered as an industrial standard. Indeed, at the end of the fermentation and maturation of lager type beers, yeast cells sediment at the bottom of tanks to form yeast slurry which represents around 2.5% of the final fermented product volume (Carmen & Ernst Ulrich, 2008). For obvious economic reasons, it is important for manufacturers to recover as much beer as possible from this slurry. Moreover, MF is emerging as a technology used for the separation of wort following the mashing step, for rough beer clarification (currently used by Heineken breweries), and for cold sterilization (i.e. to replace flash pasteurization) (Daufin et al., 2001). Elimination of heating process by the use of MF avoids the occurrence of organoleptic defects, and it is more and more used in the cool-sterilization of beer and wine (Baker, 2004). MF can be used not only to improve microbiological stability of wine, it can also ensure clarity of the product (Daufin et al., 2001). Moreover, RO can be used for concentration purposes as well as for dealcoholization. In contrast, it can be used to increase alcohol content (Girard et al., 2000). However, RO of wine can produce a concentrate with excessive malic acid, leading to taste faults. Recently, membrane processes have been employed for beer decolorization in the production of clear alcohol to be used in beer-based drink premixes (Carmen & Ernst Ulrich, 2008).

### 3.4.2 Other membrane-based processes

#### 3.4.2.1 Electrodialysis (electrically assisted processes)

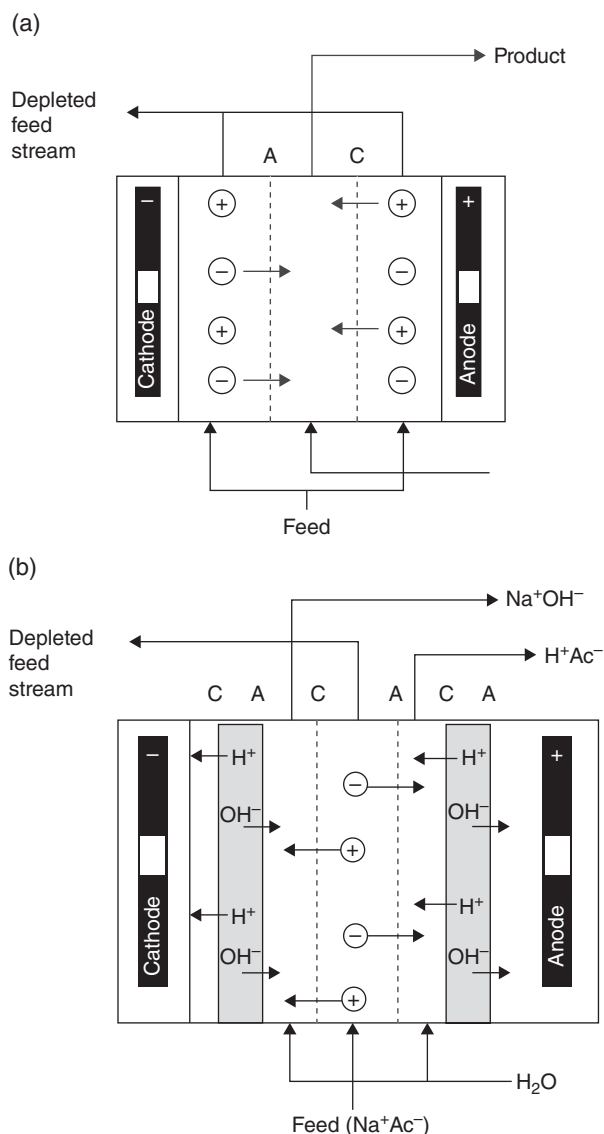
Electrodialysis (ED) is a membrane-type electrochemical process whereby membranes are used to separate ionic molecules from non-ionic ones using electric field as the driving force for separation. ED membranes are made of porous (i.e. nanometer range) ion exchange resins which selectively separate anion and cation species for dilution/concentration applications. In ED processes,

anion-permeable or “anionic” membranes are made of resin bearing fixed cationic groups, while cation-permeable or “cationic” membranes are made of resin bearing fixed anionic groups. In practice, anionic and cationic membranes are arranged alternately and are separated by spacers to form thin compartments (the stack unit). Each compartment is either for dilution or for concentration purposes, and electrodes are placed at both ends of the system (Figure 3.19). In the industry, 100–200 membranes may be disposed to form a stack unit, and industrial ED systems are composed of one or more stack units. By the application of an electric field, anion species permeate throughout the anionic membranes in the opposite direction to the electric current while cations migrate, in the same direction as the electric current, throughout the cationic membranes (Brennan & Grandison, 2012).

In a simplified version of an ED system illustrated in Figure 3.19a, anions move towards the anode and exit from the left compartment by passing through an anionic membrane, as a cationic membrane keeps them in the center compartment. In the same way, cations leave the right compartment by passing through a cationic membrane but are trapped in the center compartment because of the repulsion of the anionic membrane. The result is that, simultaneously, the pumped feed is ion depleted and the final product recovered from the adjacent compartments, called the concentration compartments, is ion-enriched (Munir, 2006). This process has the singularity of leaving the concentration in dissolved non-ionic compounds unchanged. Indeed, even if proteins or other charged macromolecules are attracted by the electric field, their high molecular weights do not allow for permeation throughout the membranes.

Bipolar-membrane ED uses special multilayer membranes to dissociate water in  $H^+$  and  $OH^-$  ions as an electric field is applied. Bipolar membranes are composed of three layers: (1) an anion exchange layer; (2) a hydrophilic interface; and (3) a cation exchange layer (Bazinet et al., 1998). As illustrated in Figure 3.19b, when those membranes are added to a conventional ED unit, it allows for the conversion of a neutral salt stream to an acid and base one. This technology is of particular interest to food processing if the product to be treated is an acid or a base.

The main application of ED in the food industry is the desalination of cheese whey, but it can also be used to concentrate, purify, or modify foods (Bazinet et al., 1998). ED applications include the removal of calcium from milk and lactic acid from whey, the control of sugar/acid ratio in wine, the pH control of fruit juices and fermentation reactors, and the purification of bioactive peptides.



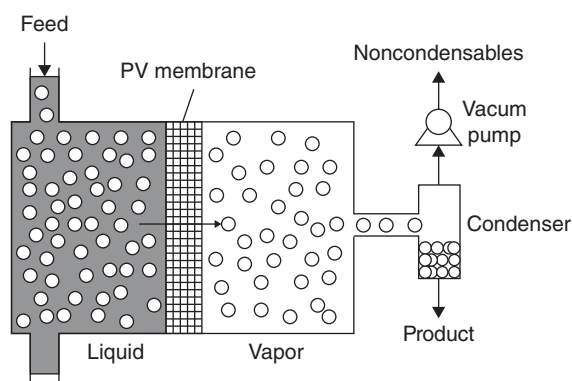
**Figure 3.19** Schematic diagrams of (a) conventional electrodiagnosis unit and (b) bipolar electrodiagnosis unit (Munir, 2006).

As for all the other membrane processes, ED membranes are susceptible to polarization and fouling and thus, the maximum salt removal attainable by this method is around 90% (Brennan & Grandison, 2012).

### 3.4.2.2 Pervaporation

Pervaporation is a relatively new membrane separation process and represents one of the most active research





**Figure 3.20** Schematic diagram of pervaporation of a two-component system (Munir, 2006).

areas in membrane technology. Unlike MF, UF, NF, RO, and ED, pervaporation is a pressure-driven membrane where the permeate generated is a low-pressure vapor, not a liquid, that can be condensed and collected or further concentrated (Feng & Huang, 1997). Using pervaporation, feed containing volatile components is fractionated when it partially vaporizes and passes through a dense or non-porous selective membrane (Karlsson & Tragardh, 1996). The driving force for separation is the chemical gradient across the membrane, but a vacuum is commonly applied on the permeate side to create an artificial pressure gradient, as shown in Figure 3.20.

Pervaporation is considered as a three-step process: (1) absorption, (2) diffusion, and (3) evaporation (or desorption). In the solution stage, the vaporizing component is drawn into the membrane at the upstream interface by chemical affinity. It then diffuses through the membrane and desorbs from the downstream interface as the membrane selectively allows for the desired components to permeate through, as vapor. The partial vaporization of the feed through the membrane is responsible for the separation ability of the pervaporation (Karlsson & Tragardh, 1996). Since the chemical composition of the membrane determines which components will permeate, a hydrophilic membrane can thus be used to dehydrate organic solutions, while a hydrophobic membrane is suitable for extracting organic components from aqueous solutions.

Pervaporation is a useful alternative for liquid mixtures that are difficult to separate by distillation and is commercially used for alcohol dehydration. In the food industry, pervaporation can be used to concentrate fruit

juices, alcohol in fermentation broth, dealcoholization of alcoholic beverages (i.e. final product of 0.5% v/v of ethanol), and recovery and concentration of aroma compounds (Karlsson & Tragardh, 1996). The latter is probably the most promising application of pervaporation in food processing. Conventional concentration processes such as evaporation, inevitably result in aroma compound losses, due to their highly volatile nature. Pervaporation can be used on raw materials, prior to evaporation to concentrate aroma (i.e. at least 100-fold greater than in the raw material), or on the resulting stream to recover lost aroma. Aroma compounds can then be reintroduced in the final product to ensure taste acceptability (Karlsson & Tragardh, 1996). Compared to other aroma recovery processes, pervaporation operates at low temperature (i.e. 20°C), which eliminates damage due to high heat and allows for energy savings.

In the dealcoholization application of pervaporation, an important concern is the loss of aroma compounds in the ethanol phase – due to their very hydrophobic nature. However, new techniques using pervaporation allow for the production of 0.5% (v/v) alcohol wine with more than 80% aroma retention (Karlsson & Tragardh, 1996). Pervaporation has also been proposed as a deodorization process for food industry effluents. It simultaneously allows the recovery, the concentration, and the valorization of flavor compounds from industrial wastes (Galanakis, 2012). However, pervaporation is in a less advanced state than all other membrane processes presented in this section, and understanding of the separation mechanisms is still incomplete. Further research is needed before large-scale pervaporation can be widely promoted (Feng & Huang, 1997).

### 3.5 Sustainability of separation technologies in food processing

In food industry separation technologies, sustainability problems are mainly related to two factors: (1) energy, and (2) water consumption. As noted by Muralidhara (2010), of the 100 quads (1 quad =  $10^{15}$  BTU/year) of energy consumed in the United States, food processing uses approximately 2 quads. At a cost of \$8 per  $10^6$  BTU, this amounts reaches \$16 billion per year. Half of this energy is used for concentration and drying purposes. For example, corn (wet) milling uses 93.7 trillion BTU, grain milling 153.3 trillion BTU, and vegetable oil processing 2.0 trillion BTU each year. Thus, there are opportunities for the integration of membrane technology to the food industry's separation and concentration processes in

many kinds of applications. As a purely physical separation, membrane concentration does not require a heat-producing unit, or a phase change (from liquid to vapor), and it can partially or totally replace energy-consuming processes (e.g. distillation and evaporation). It thus results in not only in major energy savings, but in higher product quality. Modern filtration units are compact, efficient, simple, highly selective (i.e. to specific compounds), reduce chemical production, and offer operation flexibility to integrate hybrid processes. They represent part of the solution to quality, energy, space, and environmental concerns (Merry, 2010). Moreover, membrane technologies allow for the re-use and recovery of all process streams, without damage to the environment.

Food processing also consumes large volumes of water which are a major concern for the environment. Advanced membrane technologies represent a solution to polluting, high-energy, space-consuming, conventional waste water complex processes (Madaeni et al., 2004). Indeed, process effluents are today being treated using membrane processes such as RO, UF, and ED and treated effluents are being reused in plants for different purposes such as washing.

In conclusion, membrane technologies have a promising future and may be considered as important tools for the efficient use of processing streams without polluting discharges with regard to energy savings, environmental, and quality regulations.

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