3 Centrifugation–Filtration

Theodoros Varzakas

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3.1 INTRODUCTION

Centrifuges are used to clarify nonsettling solutions containing finely divided solids; to break emulsions of two immiscible liquids; to obtain a clear solution from a liquid–solid mixture; or to separate two immiscible liquids.

Separation operations involve properties such as particle size, density, and solubility, and have to do with the separation of solids from solids, solids from liquids, liquids from liquids, and solids from gas.

Examples of separation of solids from solids with regard to particle size include size grading of fruits and vegetables, and screening of wheat, tea, sugar, and cocoa beans.

On the basis of particle size and density, we have the phenomenon of settlement, that is, suspension of solid in fluid, liquid, or gas and typically, the finer solids (such as kaolin) of smaller size and/or density in the feed slurry are separated in the centrate stream as product (e.g., 90 percent of particles less than 1 mm, etc.), whereas the larger and/or denser solids are captured in cake as reject. Furthermore, separation can be in the form of thickening, where solids settle under centrifugal force to form a stream with concentrated solids (Perry, 1999). The larger and denser particles will settle faster than the smaller and less dense ones. Classification implies the sorting of particulate material into size ranges. Use can be made of the different rates of movement of particles of different sizes and densities suspended in a fluid and differentially affected by imposed forces such as gravity and centrifugal fields. Separation can also be in the form of classification and degritting at which separation is effected by means of particle size and density (Perry, 1999).

Regarding solubility, examples are the extraction of sugar from sugar beet or coffee from ground-roasted beans where one of the components is soluble.

Regarding the separation of solid from liquid on the basis of particle size and filtration, a solid suspension is forced in the liquid through a filter medium, cloth, or screen by the development of a pressure gradient across the filter medium. This is carried out in a filter press. Examples are filtration of sugar solutions during refining, and filtration of canning brines and syrups.

Regarding the particle size and density and settlement, centrifugation subjects the solid suspension in a liquid to a cyclic motion in a bowl with a perforated wall. Centrifugal force forces the liquid out through the perforations of the bowl wall. The size of perforations determines the portion of solid retained in the bowl. Examples include dewatering of sugar crystals and milk clarification.

Examples of separating liquid from liquid includes the separation of cream from milk and dewatering oils. Settlement allows the more dense liquid to be rejected. Centrifugation subjects the mixture to a cyclic motion in a solid bowl removing the more dense liquid through an outlet near the circumference of the bowl and the less dense liquid through an outlet near the center of the bowl’s rotation.

Regarding solubility examples are the crystallization of sugar from solution and extraction where one of the liquid components dissolves out as it occurs in solvent extraction of oils and flavouring materials (Butters, 1993; Loncin and Merson, 1979).

Finally, an example of the separation of a solid from a gas includes the bag filtration method of removing dried powder from the air of a spray drier on the basis of particle size and filtration.
Regarding particle size and density, cyclone separation of dried powder from the spray drier subjects the solid suspension in gas to a cyclic motion, hence separating out the more dense solids. On the basis of solubility, wet scrubbing of the powder (dissolving out the solid) occurs out of the exhaust air of a spray drier using the solvent as a feed material.

Diffusion coefficients for glucose and malic acid have been measured by the lag-time model using cell-free and cell-occupied calcium alginate membranes placed in a plate diaphragm diffusion cell (Teixeira et al., 1994). The results indicated that neither carbon dioxide release nor solute concentration or concurrent diffusion affected the diffusion coefficients of either solute. The presence of cells caused a decrease in solute transport across alginate membranes and a reduction in the diffusion coefficients. The mass transfer mechanism in cell-occupied membranes was best characterized by the random pore model, a model describing diffusion through gel membranes. This model described the influence of cell concentration on the effective diffusion coefficient for the experimental results tested. The value obtained for the diffusion coefficient of glucose was $6.6 \times 10^{-10}$ m$^2$ s$^{-1}$ and was of the same order of magnitude as the molecular diffusion coefficient of glucose in water. However, $D_{eff}$ for malic acid was found to be equal to $4.36 \times 10^{-10}$ m$^2$ s$^{-1}$ and was significantly lower than that in aqueous solution ($8 \times 10^{-10}$ m$^2$ s$^{-1}$). The lag-time analysis method has also been used to evaluate diffusion coefficients of lactose and lactic acid through a 3% agarose gel membrane (Bassi et al., 1987). Average diffusion coefficients were estimated to be $3.97 \times 10^{-10}$ and $2.9 \times 10^{-10}$ m$^2$ s$^{-1}$ for lactose and lactic acid, respectively. The effective diffusion coefficient was affected by several factors, including the shape, density, length, and diameter of the pores within and on the surface of the matrix.

A diaphragm diffusion cell has been used to evaluate the effective diffusion coefficient of ethanol in a 4% (w/v) agarose gel at 25°C. A mean value of $9.5 \times 10^{-10}$ m$^2$ s$^{-1}$ was reported by Westrin and Axelsson (1991). This value agreed with the value of the molecular diffusion coefficient of ethanol in water. Djelveh et al. (1989) also used an improved cell diffusion method based on a diaphragm cell to determine diffusion constants in gels or foods. A two-compartment diffusion cell has been used by Coca et al. (1986) to study the permeation of potassium chromate solutions through rat epidermis at 37°C. They reported a change in diffusivities with solute concentration. Partition coefficients were also concentration dependent.

### 3.2 POLYSULFONE MEMBRANE CHARACTERISTICS: DEFINITIONS, USES, STRUCTURE, AND FUNCTIONS

Membrane polymers are often chemically inert. Polysulfone (PS) is one material suited to meet this goal and possesses very good chemical, thermal, and biological stability combined with excellent mechanical strength and flexibility (Rodemann and Staude, 1995). PS membranes are microfiltration (MF) or ultrafiltration (UF), synthetic polymeric, hydrophilic membranes. They are used because of their ability to cope with wide temperature limits, wide pH tolerance (1–13), good chlorine resistance, wide range of pore sizes (10–200 Å), and molecular weight cut-off (MWCO) ranging between 1000 and 500,000. Their only limitation is the restriction to low
pressure drops. They are characterized by having diphenylene sulfone repeating units in their structure. The \( \text{SO}_2 \) group in the polymeric sulfone is very stable because of the electronic attraction of resonating electrons between adjacent aromatic groups. The oxygen molecules projecting from this group each have two pairs of unshared electrons to donate to strong hydrogen bonding with solute or solvent molecules. Repeating phenylene rings create both steric hindrance to rotation within the molecule and electronic attraction of resonating electron systems between adjacent molecules; both contribute to a high degree of molecular immobility, producing high rigidity, strength, creep resistance, dimensional stability, and heat deflection temperature. Phenyl sulfone groups have long-term high-temperature stability (Mulder, 1991).

An Intersep Nadir PS membrane (Intersep Filtration Systems, Wokingham, Berkshire) was used in these experiments. This membrane was polypropylene backed for improved handling and repeated use with a skin modified by polyvinyl dipuridone to be hydrophilic. The industrial production process employed in the manufacture of this membrane offers controlled retention characteristics, water permeability, and solute transport. Such asymmetric membranes consist of a thin top layer supported by a porous sublayer with the resistance to mass transfer being almost completely determined by the top layer. Therefore, \( L \), the effective thickness of the membrane, has been taken to be approximately equal to \( 0.05 \text{ mm} + 0.25 \mu \text{m} = 0.05 \text{ mm} = 50 \mu \text{m} \).

The porous sublayer thickness was not taken into account for the calculation of the effective thickness of the membrane since it was very small.

Other important properties show that it is inert, noncytotoxic, and does not denature biological materials; also, it has broad chemical resistance and wide pH range compatibility. Its nominal MWCO is 100 kDa, implying that solutes with a MW greater than 100,000 are rejected more than 90%. According to Intersep’s membrane catalog, the PS membrane shows less than 10% rejection for bovine serum albumin (BSA), which has a MW of 67 kDa. It has been assumed that the rejection for lysozyme and cellulase is approximately zero. Typically, the membranes may be reused 10 times in laboratory stirred cells and withstand over a year of constant use in the cartridge configuration.

The membrane cleaning procedure followed was the following:

- Flushing with warm water
- Cleaning with 0.5% P3-ultrasil 10
- Flushing with water
- Cleaning with 0.5% P3-ultrasil 75
- Flushing with water
- Cleaning with 1% P3-ultrasil 10
- Flushing with water
- Sanitizing with 1% P3-ultrasil 25

This hydrophilic PS membrane was washed with deionized water and equilibrated with the buffer solution for 1 h before being placed in the diffusion cell. Then it was left in the buffer for another hour at stirring conditions.
3.2.1 Polysulfone Membrane

According to the membrane catalog provided by Intersep, the permeability water flux for the PS membrane is 450 LMH (L m\(^{-2}\) h\(^{-1}\)) tested at 3 bar, 700 RPM, 20°C, stirred cell.

The pore geometry is also very important in the permeability method and needs to be determined so that the experimental results could be interpreted. Therefore, assuming cylindrical, perpendicular pores, which imply tortuosity equal to unity (T = 1) and assuming that all pores have the same radius, using the Hagen–Poiseuille equation as described in Mulder (1991), the radius of the pores could be estimated as follows:

\[
J = \frac{(\varepsilon r^2 / 8 \mu T)}{L} \cdot (\Delta P / L) \quad (3.1)
\]

where J is the volume flux through the pores equal to \(450 \times 10^{-3} \text{ m}^3/3600 \text{ m}^2 \text{ s}\); \(\varepsilon\) is the surface porosity, which is equal to the ratio of the effective area (S) to the area of the membrane (A), and hence \(\varepsilon = (2 \times 10^{-5} \text{ m}^2)/(4(\pi / 4)(4 \times 10^{-2})^2 \text{ m}^2) = 0.0159\) and usually has a low value ranging from 0.1% to 1% for UF membranes; \(\mu\) is the viscosity and is equal to \(10^{-3} \text{ N s m}^{-2}\); \(T\) is the tortuosity equal to unity; \(\Delta P\) is the pressure difference across the membrane and is equal to \(3 \times 10^5 \text{ N m}^{-2}\); and \(L\) is the thickness equal to \(50 \times 10^{-6} \text{ m}\). This equation indicates that the solvent flux is proportional to the driving force, that is, the pressure difference across a membrane of thickness \(L\) and inversely proportional to the viscosity.

By substituting these data in Equation 3.1, it is evident that the average value of the radius of the pores is 102 nm. In UF membranes, the average pore diameter (i.e., diameter of pore in the dense skin) is generally in the range of 2–100 nm. The Hagen–Poiseuille equation gives a good description of transport through membranes consisting of a number of parallel pores. However, very few membranes have such a structure in practice.

Insulin transport phenomena across a series of porous charged membranes were studied at two pH conditions (pH 3.3 and pH 7.4) by Zhang et al. (2009). The membranes were prepared by pore-surface modification of porous poly(acrylonitrile) (PAN) membranes by grafting with weak acidic and basic functional groups. The insulin partition coefficient \(K\) between the membrane and the solution was estimated from the equilibrium adsorption amount in the batch adsorption experiment. The insulin-effective diffusion coefficient \(D\) inside the membrane was determined as a fitting parameter by matching the diffusion model with the experimental data of the diffusion measurement. Both \(K\) and \(D\) correlated well with the charge properties of the insulin and membrane. When the insulin and membrane carried opposite net charge, the partition coefficient showed relatively larger values, while the effective diffusion coefficient was reduced. The insulin permeability coefficient \(P\) obtained from the experimental results agreed with that estimated from the partition coefficient and effective diffusion coefficient. These results suggested that the combined effects of the solubility and diffusivity on the permeability coefficient complicated the relationship between the permeability and the charged properties of the insulin.
and membrane. Moreover, insulin permeability was reduced by the boundary layer between the membrane and solution (Varzakas, 1998).

### 3.3 CHARACTERISTICS OF MEMBRANES: MASS TRANSFER IN MEMBRANE TECHNOLOGY

For the separation of macromolecules, pressure could be applied depending on the membranes used or the difference in electric potential. The last case refers to electrodialysis (ED), which is an electrochemical process in which a direct electric current causes ions to move from a less concentrated solution to a more concentrated solution. Anion (negative) and cation (positive) ions transfer through selective membranes (Figure 3.1).

In conventional ED, flat-sheet membranes are stacked in alternating layers of cation-exchange and anion-exchange membranes. With the application of electrical current, cations migrate through the cation-exchange membranes toward the cathode, but the cations are stopped by anion-exchange membranes. Similarly, anions migrate toward the anode through anion-exchange membranes but are stopped by cation-exchange membranes. An extension of conventional ED uses bipolar membranes, which splits water into its component $\text{H}^+$ and $\text{OH}^-$ ions. When used along with conventional cation- and anion-exchange membranes, it allows a salt stream to be converted into an acid and a base stream. This is particularly useful in downstream processing of organic acids such as citric, lactic, acetic, and gluconic acids, in that it produces the more desirable acid form of the compound while regenerating the alkali, which is used in the fermentation vessel (Cheryan, 2007).

Like other membrane processes, ED is affected by concentration polarization and membrane fouling. In addition, ED also exhibits simultaneous water transport,
which limits the maximum concentration that can be attained for permeable species. With bipolar membranes, fouling by divalent cations is quite severe, since they can precipitate with the hydroxyls encountered in the cationic membranes. ED is generally more expensive than pressure-driven membrane processes, primarily due to the electrical energy requirements. It is justified in some applications, such as water desalting, tartarate removal from wine, demineralization of protein solutions, and separation and concentration of organic acids (Bailly et al., 2001).

Pervaporation is also a pressure-driven process except that unlike all the others discussed so far, the permeate is a vapor and not a liquid. The solutes permeating the membrane are relatively more volatile than the solvent.

The driving force for transport is a chemical potential gradient that arises due to a decrease in the activity of the permeating components. The activity decrease can be accomplished by pressure reduction, for example, a vacuum can be applied on the permeate side. The vapor is then condensed and the noncondensables are removed by the vacuum pump (Rajagopalan et al., 1994; Rajagopalan and Cheryan, 1995).

Process fluid passing through the membrane is named permeate, whereas fluid that does not pass through is called (concentrate-retentate) (Walstra et al., 1999). The schematic diagram of flow through a membrane system is shown in Figure 3.2.

The same phenomenon occurs during the operation of cross-flow filtration. In a cross-flow ultrafilter used for the concentration or separation of macromolecules, process fluid is passed over a filtration membrane. The pressure being higher in the feed side, there is a flow of permeate through the membrane and the retentate leaves the membrane system since it cannot pass through the membrane being more concentrated in species.

If one considers what happens to a protein being unable to pass through the membrane and assumes that the species concentration in the bulk stream is $C_b$ and that the flow rate across the membrane is high then the bulk concentration remains constant in the membrane (Pyle, 1992). We also assume that the stream flowing across the membrane comprises a well-mixed turbulent core and a thin boundary layer. Then the flux $J$ is $Q/A$, where $Q$ is the flow rate of the permeate and $A$ is the membrane area. The flux of permeate is expected to follow Darcy’s law and hence

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\[ J = \Delta P/Wm, \]  
where \( \Delta P \) is the pressure drop and \( Wm \) the hydraulic resistance of the membrane depending on its pore size distribution, voidage fraction, and viscosity of the permeate.

If the protein cannot pass through the membrane, it will accumulate in the boundary film. Hence, concentration increases toward the membrane surface, and protein, due to Fick’s law, will diffuse back toward the bulk. If a steady state is established within the boundary layer and no net transfer toward the membrane occurs, then the bulk flux and the back-diffusional flux must be equal (Pyle, 1996). This concentration increase toward the membrane surface is called concentration polarization. Owing to the transport through the membrane of molecules/particles below a certain size, molecules/particles above this size are concentrated on the feed side of the membrane surface (Gekas and Hallström, 1987; Hallström et al., 2007). It should also be noted that the mean driving force over the boundary layer is the log-mean concentration difference.

Finally, a negative feature of polarized membranes is when permeate flux becomes independent of the applied pressure drop.

Depending on the type of membrane, the size of the membrane pores, the applied pressure, and the type of particles retained (Figure 3.3), we have the following filtration operations: reverse osmosis (RO), NF, UF, MF, and ED. All these have applications in the food and drink industries.

3.3.1 Membrane Technology and Its Uses

Membrane technology is useful for the selective enrichment of some ingredients. For the manufacture of yogurt, different technologies can be employed such as RO, NF, UF, and MF in order to increase the solids by removing water. Their use is limited in the evaporation of skimmed milk, which will be the raw material for the manufacture of different types of yogurt. Some of these methods remove part of lactose and inorganic salts of milk with the result of increasing the protein content. These technologies can be used to evaporate skimmed milk containing a maximum of 9–12% soluble solids. In the retentate, enough lactose remains to facilitate fermentations (Kilara, 2006).

Membrane applications in conventional food processing technologies are well described (Cuperus, 1998; Cuperus and Nijhuis, 1993), involving dairy (Daufin et al., 2001; Rosenberg, 1995; Zydny, 1998), fruit juice and beverages (Girard and Fukumoto, 2000; Jiao et al., 2004), fats and oils (Bhosle and Subramanian, 2005; Koseoglu and Engelgau, 1990; Koseoglu 1991; Manjula and Subramanian, 2006; Snape and Nakajima, 1996), and starch (Rausch, 2002; Singh and Cheryan, 1998).

Membrane applications of lipid-, carbohydrate-, and protein-based nutraceuticals and some minor bioactive components have been critically evaluated by Akin et al. (2012). Both nonporous and porous membranes were employed for lipid-based nutraceutical separation. The use of nonporous membranes together with nonaqueous solvents brought about the impact of solution-diffusion theory on transport through membranes. Both organic and inorganic membranes gave encouraging results for the recovery of lipid components with single- and/or multistage membrane processing. Two-stage UF–NF systems with polymeric membranes provided an efficient approach for the removal of high- and low-MW unwanted components, resulting in higher-purity oligosaccharides in the NF retentate. The charged nature of protein-based nutraceutical components had a major effect on their separation. Operating at optimal pH levels was critical for fractionation, especially for low-MW peptide hydrolysates. Processing of minor components such as polyphenols utilized all types of porous membranes from prefiltration to concentration stages. The coupling of membrane separation and supercritical fluid technologies would combine unique advantages of each process, resulting in a novel separation technology offering great potential for the nutraceutical and functional food industry.

3.3.1.1 Ultrafiltration

UF can be used to concentrate molecules such as peptides, proteins, or other particles. Membranes have been constructed in such a way as to allow molecules to pass until a certain MW and a pore size of approximately 0.1 μm (Rosenberg, 1995). The pressure applied in ultrafiltration ranges between 1 and 6 bar resulting in concentration of molecules of MW 1000–50000 Da. Moreover, the supply rate in UF is high with the aim to prevent the obstruction of the membrane pores. The construction material in these membranes is cellulose with derivatives such as cellulose acetate (CA) and some thermotolerant polymers such as polysulphone and polyethersulphone.

UF separates effectively macromolecules (proteins) and particles (casein micelles, fat cells, somatic cells, and bacteria) of milk. The main aim of ultrafiltration is the
increase in protein concentration and is applied in skimmed milk and whey. Initially, it could cause significant changes in the composition of dairy products manufactured with ultrafiltrated milk and for this reason, it allows the production of novel products (Walstra et al., 1999).

UF is applied in skimmed milk and whey for the manufacture of fresh and feta cheeses, since it leads to an increase in the yield, decrease in the quantity of added rennet, and a better quality of the produced cheese. It is used in the preconcentration of milk, fractionation of whey, and micellar casein enrichment for cheese production, because of the low cost, energy efficiency, and consistent product quality.

UF can also be used in the manufacture of yogurt. Following UF, milk presents increased solids due to the concentration of its macromolecules (lipids and proteins) (Kilara, 2006). These yogurts have a better texture, higher cohesion, gentle aroma, and pleasant taste. They have an increased concentration of proteins—up to 50%—and low concentration in lactose—approximately 50%—compared to commercial-type yogurts, whereas they have increased calcium and iron concentration, which give specific characteristics and high added value to the product (Rinaldoni et al., 2009).

Cross-flow filtration in the final steps provides important advantages to designed processes since membrane polarization is reduced when compared to plug-flow filtration. Transmembrane pressure is relatively constant and high-throughput operations can be implemented with proper industrial equipment (Ghosh and Cui, 2000).

One of the most attractive segments in the food and cosmetic industries is that of natural pigments. Natural pigments obtained through biotechnological processes represent an attractive alternative. Ruiz-Ruiz et al. (2013) has previously worked on the development of an aqueous two-phase system (ATPS)-based prototype process for the recovery of B-phycoerythrin (BPE), a natural high-value pigment obtained from *Porphyridium cruentum*.

Detailed studies describing the scaling up of ATPS processes from bench scale to pilot plant facilities are not common. They described experiences derived from the scale-up of a previously developed process for production and recovery of highly purified (purity defined as the absorbance ratio $A_{545}/A_{280} > 4$) BPE, where a scale-up factor of 850 was implemented. Characterization of cell disruption with a pilot-scale bead mill allowed efficient BPE release at 2900 rpm, 10% (w/v) sample load, 60% (v/v) bead load, and 0.5 mm glass beads and 22 min of residence time with a yield of 1.35 mg BPE g$^{-1}$ of wet biomass.

BPE was recovered and purified using a strategy comprising isoelectric precipitation, aqueous two-phase fractionation and UF. A 54% global BPE recovery yield, with final purity of 4.1, was achieved under optimal process conditions. Considering total costs for raw materials and energy expenditures for one batch, it was determined that the production cost of BPE was $1.17 USD mg$^{-1}$, which is less than the commercial price of a BPE standard (>30 USD mg$^{-1}$).

Liquid–liquid extraction systems such as ATPS allow rapid recuperation and purification of biological products, integration and intensification of bioprocesses, development of a biocompatible medium for biologic compounds and scale-up feasibility (Rito-Palomares, 2004; Benavides and Rito-Palomares, 2008).
Yokota et al. (2012) has developed a novel analytical method for the quantification of bromate in fresh foods using high-performance liquid chromatography (HPLC) with a postcolumn reaction. The fresh food sample solutions were pretreated with homogenization, centrifugal UF, and subsequent solid-phase extraction using a strong anion-exchange resin. After separation on a strong anion-exchange chromatography column using a highly concentrated NaCl solution (0.3 M) as the eluent, the bromate was quantified by detection using a postcolumn reaction with a noncarcinogenic reagent (tetramethylbenzidine). The developed HPLC technique made it possible to quantify bromate in salt-rich fresh foods. The recoveries from fresh foods spiked with bromate at low levels (2 or 10 ng g\(^{-1}\)) satisfactorily ranged from 75.3% to 90.7%.

The lowest quantification limit in fresh foods was estimated to be 0.6 ng/g as bromic acid. The method should be helpful for the quantification of bromate in fresh foods disinfected with hypochlorite solutions.

Solid–liquid separation is a very important unit operation related to chemical, engineering, and environmental processes. A number of conventional separation methods and hardware, such as centrifugation, filtration, or sedimentation, have been in use for a long time (Rossignol et al., 2000; Sharma, 1994). However, nowadays, we are looking for techniques and processes with less energy and capital.

Biomimetics is a technique by which phenomena in nature are used as the basis to modify existing technologies or design new ones (Bulger et al., 2008). Hung et al. (2012) developed a new particle separator based on the mechanism of cross-flow filtration employed by suspension-feeding fish. When water enters a suspension-feeding fish’s oral cavity, it brings in food particles suspended in large quantities of water. A high proportion of the particles that enter are retained inside the fish’s oral cavity and eventually swallowed while most of the water exits via branchial slits.

To construct the model of the bioinspired particle separator, computational fluid dynamics techniques are used, and parameters related to separator shape, fluid flow, and particle properties that might affect the performance in removing particles from the flow are varied and tested. The goal is to induce a flow rotation, which enhances the separation of particles from the flow, reduce the particle-laden flow that exits via a collection zone at the lower/posterior end of the separator, while at the same time increase the concentration of particles in that flow. Based on preliminary particle removal efficiency tests, an exiting flow through the collection zone of about 8% of the influent flow rate is selected for all the performance tests of the separator, including trials with particles carried by air flow instead of water. Under this condition, the simulation results yield similar particle removal efficiencies in water and air, but with different particle properties. Particle removal efficiencies (percentage of influent particles that exit through the collection zone) were determined for particles ranging in size from 1 to 1500 μm with a density between 1000 and 1150 kg m\(^{-3}\) in water and 2 and 19 mm and 68 and 2150 kg m\(^{-3}\) in air.

In UF of milk, nonprotein nitrogen and soluble components such as lactose, salts, and some vitamins pass through the membrane, whereas milk fat, proteins, and insoluble salts are retained by the membrane (Mehaia, 1997). The growing use of UF in the dairy industry, especially in the area of value addition, promises to dramatically change the technology of concentrated and dried milk products. Its main
advantages are the higher dry matter and milk protein contents and the increased ratio of protein to dry matter compared to native milk.

Dairy whitener is widely used as a substitute for fresh milk, cream, or evaporated milk in tea, coffee, cocoa, or drinking chocolate and is also suitable for adding to foods like soups, sauces, puddings, and cereal dishes. The main advantages of using dairy whitener are ease of handling, improved shelf life, which may be specific requirements for use in restaurants, railways, airways, and waterways (Khatkar et al., 2012a).

Dairy whiteners should also have the ability to withstand the high temperature (80–90°C) and low pH (4.6–5.2) of coffee solution (Khatkar et al., 2012a).

The whitening effect is produced in coffee as a result of light scattered from the surface of finely emulsified particles. The whitening powder comes mainly from a well-emulsified and finely dispersed fat and protein in a colloidal state (Khatkar et al., 2012b).

Khatkar et al. (2013) studied the physicochemical and functional quality attributes of dairy whitener prepared from a UF process. Developed dairy whitener had significantly ($P < 0.01$) greater protein (40.07 ± 0.66%) and calcium (1.42 ± 0.05%) contents compared to market samples and also had good solubility index (0.25 mL) and significantly ($P < 0.01$) higher dispersibility (92.08%) and $L^*$ value (93.87). Dairy whitener, even at lower solids level, had an edge over the market samples in terms of sensory and instrumental color characteristics in both tea and coffee without leaving any undissolved suspended particles. Dairy processors would be able to prepare value added dairy whitener commercially that would increase their profitability.

Recovery of high value-added products such as proteins, aromas, and flavors can be done using UF processes (Vandajon et al., 2002). The application of membrane technology as the main method of separation, concentration, and purification (Murado et al., 2010) of valuable compounds from industrial waste materials has been applied to diverse sources, including fish meal (Afonso et al., 2004), palm oil mill effluents (Wu et al., 2007), and solid by-products of the brewing industry (Tang et al., 2009).

Rodriguez-Amado et al. (2013) focused on the production of antihypertensive and antioxidant activities using enzymatic hydrolysis of protein concentrates recovered by UF of different wastewaters from the industrial processing of cuttlefish (*Illex argentinus*). The effluents were produced in the processes of thawing (E1), softening (E2), boiling (E3), and gelation (E4). Their results showed that membranes with cutoff at 100, 30, and 10 kDa were an effective resource to protein concentration of E2 and E3 but limited for E1 and E4. In addition, E2 and E3 retentates led to remarkable antihypertensive and antioxidant activities, further improved by enzymatic hydrolysis. Also, sequential UF revealed the enrichment of these protein concentrates in peptides with high angiotensin-converting enzyme (ACE)-inhibitory activity.

Thereby, UF-fractionation followed by proteolysis of protein concentrates from cuttlefish wastewaters offers new opportunities for the development of bioactive hydrolysates with application in the food industry. In addition, this approach contributes to an improved depuration of industrial wastewaters, reducing the treatment costs and leading to a decrease in its contaminating effect.

Different cascades of UF–DF were employed using waste effluents. For this purpose, Prep/Scale-TFF cartridges (Millipore Corporation, Bedford, MA, USA)
of 100, 30, 10, and 1 kDa MWCO were used by Rodriguez-Amado et al. (2013). According to the manufacturer, cartridges were made of polyethersulfone (PES), except for 1 kDa, which was from regenerated cellulose.

The operation mode was the following: an initial phase of UF at 40°C with total recirculation of retentate was performed, immediately followed by a DF step. During UF, the inlet pressure remained constant to determine the drops of flow rate due to the increased concentration of the retentate and possible adhesions to the membrane.

Castel et al. (2012) compared protein yield, protein concentration, and physicochemical characteristics of *Amaranth mantegazzianus* protein concentrates (APCs) obtained at pilot scale by a conventional process (CP) (alkaline extraction and isoelectric precipitation) and two alternative processes (AP): (1) acid pretreatment process combined with isoelectric precipitation and (2) acid pretreatment process combined with UF. Although AP resulted in higher protein concentration, protein yield was lower than in CP. SDS–PAGE and size-exclusion chromatography showed high-molecular-weight fractions only for isoelectric precipitation concentrates (obtained by CP and AP). The amino acids concentration, especially phenylalanine, isoleucine, and methionine, increased in all protein concentrates with respect to the amaranth flour. Particularly, the product obtained by UF was rich in phenylalanine and lysine, and presented no limiting amino acid with respect to the recommendation of the Food and Agriculture Organization of the United Nations (FAO). In conclusion, process (2) improved protein concentration and nutritional quality (balanced amino acid composition) of APCs with respect to CP and process (1), suggesting that the UF process is a viable alternative to CP and a promising method for obtaining protein concentrates.

Simultaneously, several biomass-based processes were developed over the past decade suggesting scenarios from a classic biofuel plant to a new biorefinery concept, which produces, for instance, polymers that were previous fossil resource based. The growth of bioresource-based chemicals, functional monomers, as well as fuels, leads to an increased demand for new separation processes. This review by Abels et al. (2013) highlights the role of membrane separations within current and future biofuel and biorefinery scenarios. Membrane processes reviewed are, for instance, pervaporation for alcohol recovery and UF of canola oil, as well as new developments such as the UF/NF of lignin in a solvent-based lignocellulose conversion process, or the recovery of amino acids via ED. The membrane processes are classically categorized as concentration-driven, pressure-driven, electrical-driven, and prospective. It follows the transition of a classic biofuel production plant to a new sophisticated biorefinery. The review closes with a reflection of membrane-based downstream processes required in a biorefinery, transforming cellulose into itaconic acid.

Parés et al. (2012) tested the use of serum from porcine blood as functional ingredient in frankfurter production. Three pilot productions of sausages were carried out to compare serum containing frankfurters and sausages, based on a standard commercial formula that included caseinate and polyphosphate. Both products were very similar for proximate composition, water-holding capacity, cooking and purge losses, instrumental texture, and microstructure. The sensory descriptive profile and the overall acceptance were also comparatively evaluated. Although significantly higher values for the animal taste and odor attributes of sausages with serum compared
to control ones were obtained, the differences were lower than those reported in a previous study using whole plasma. Thus, UF could be useful to reduce animal off-flavor in blood-based protein ingredients. Moreover, overall acceptance did not significantly differ between the two types of products, being 6.7 and 6.5, for control and test sausages, respectively.

Kawa-Rygielska et al. (2013) investigated the feasibility of the concentrate obtained after membrane UF of sugar beet thin juice for ethanol production and selection of fermentation conditions (yeast strain and media supplementation). The resulting concentrate was subjected to batch ethanol fermentation using two strains of *Saccharomyces cerevisiae* (Ethanol Red and Safdistill C-70). The effect of different forms of media supplementation (mineral salts: (NH$_4$)$_2$SO$_4$, K$_2$HPO$_4$, MgCl$_2$; urea + Mg$_3$(PO$_4$)$_2$, and yeast extract) on the fermentation course was also studied. It was stated that sugar beet juice concentrate is suitable for ethanol production yielding, depending on the yeast strain, ca. 85–87 g L$^{-1}$ ethanol with ca. 82% practical yield and more than 95% of sugar consumption after 72 h of fermentation. Nutrient enrichment further increased ethanol yield. The best results were obtained for media supplemented with urea + Mg$_3$(PO$_4$)$_2$ yielding 91.16–92.06 g L$^{-1}$ ethanol with practical yield ranging 84.78–85.62% and full sugar consumption.

A novel UF membrane with controllable selectivity for protein separation was obtained by Li et al. (2013) by altering the structure of the thick sieving layer on the membrane surface and subsurface. Poly(vinyl pyrrolidone) (PVP) was first cross-linked on/in the poly(vinylidene fluoride) (PVDF) hollow fiber MF membrane to attract more sulfobetaine (SB) monomer adjacent to membrane for subsequent grafting polymerization and the formed thick sulfobetaine polymer (PSB) layer on the membrane surface and subsurface acted as the sieving layer with environment sensitivity. After immersed in 20 mmol L$^{-1}$ NaCl solution at 60°C, the novel UF membrane with the sieving layer of 4.8 μm showed high permeate capacity with a water flux of 590 L m$^{-2}$ h$^{-1}$ and a good selective behavior with MWCO of 95–110 kDa. The protein mixture (BSA and Lys) could be separated through the novel UF membrane efficiently by isoelectric focusing of one component with a larger size (BSA). By means of the simple immersion in pure water, the membrane permeated mostly proteins and the degree of flux decline reduced obviously. After the membrane is swelled in NaCl solution again, the membrane restored the selectivity and the protein separation efficiency. Such smart UF membranes are attractive candidates for the batch separation of protein mixtures, expanding the membrane application in the fields of agrofood, biomedicine, and other biofiltration.

The antioxidant properties of barley glutelin hydrolysates were evaluated by Xia et al. (2012) based on their radical-scavenging capacity (DPPH/O$_2^\cdot$/OH$^\cdot$), Fe$^{2+}$-chelating effect, and reducing power. Alcalase hydrolysates (AH) demonstrated significantly higher antioxidant capacity than those treated by flavorzyme in most of the assays. The AH was separated using ultrafiltration and reversed-phase chromatography, and assessment of the fractions indicated that the large-sized peptides (Mw > 10 kDa) possessed stronger DPPH-scavenging activity and reducing power, whereas small-sized peptides (Mw < 1 kDa) were more effective in Fe$^{2+}$-chelating and OH$^\cdot$-scavenging effect. The hydrophobic fraction contributed more to Fe$^{2+}$-chelating and OH$^\cdot$-scavenging activity. Four peptides contributing to antioxidant activities were
Pro–Leu. Compared to the positive controls, AH exhibited excellent Fe\(^{2+}\)-chelating
activity and strong DPPH/OH\(^{-}\)-scavenging effect. Thus, hydrolyzed barley glutelin
is a potential source of antioxidant peptides for food and nutraceutical applications.

In the food processing industry, shrimp shells (Parapenaeus longorostris) have
great commercial value because they are rich in chitin (24 wt%), protein (40 wt%),
lipids, pigments, and flavor compounds. Benhabiles et al. (2013) examined protein
recovery by UF during isolation of chitin from shrimp shell. Up to 96 wt% of the
proteins could be removed (i.e., deproteinization) from the shrimp shells by incu-
bating them in NaOH (2 N) over 2 h, at T = 45°C, and solid to solvent ratio of 1:2
(w/v). A solute rejection coefficient (R\(_0\)) of 97% was obtained in the UF process to
recover proteins from deproteinized shell waste water. The protein concentration
process that was carried out beyond the critical flux of 380 L h\(^{-1}\) m\(^{-2}\), at a transmem-
brane pressure of 3 bars, and a tangential velocity of 5 m s\(^{-1}\) was found to reduce the
hydrolysate volume by a factor of 2.4. Owing to a reduction in organic matter in the
effluent, the chemical oxygen demand (COD) of the permeate was reduced by 87%.

The extracellular α-L-rhamnosidase has been purified by growing a new fungal
strain, Aspergillus awamori MTCC-2879, in the liquid culture growth medium con-
taining orange peel (Yadav et al., 2013). The purification procedure involved UF
using PM-10 membrane and anion-exchange chromatography on diethyl amino ethyl
cellulose. The purified enzyme gave a single protein band in SDS–PAGE analysis
corresponding to molecular mass 75.0 kDa. The native PAGE analysis of the puri-
fied enzyme also gave a single protein band, confirming the purity of the enzyme.
The \(K_m\) and \(V_{max}\) values of the enzyme for \(p\)-nitrophenyl-α-L-rhamnopyranoside
were 0.62 mM and 27.06 μmol min\(^{-1}\) mg\(^{-1}\), respectively, yielding \(k_{cat}\) and \(k_{cat}/K_m\) val-
ues 39.90 s\(^{-1}\) and 54.70 mM\(^{-1}\) s\(^{-1}\), respectively. The enzyme had an optimum pH of
7.0 and an optimum temperature of 60°C. The activation energy for the thermal
denaturation of the enzyme was 35.65 kJ mol\(^{-1}\) K\(^{-1}\). The purified enzyme can be
used for specifically cleaving terminal α-L-rhamnose from the natural glycosides,
thereby contributing to the preparation of pharmaceutically important compounds
like prunin and L-rhamnose.

UF experiments of polysaccharide macromolecule have been performed in a
batch, a stirred as well as unstirred membrane cell using a fully retentive membrane
over a wide range of operating conditions as described by Sarkar (2013).

A model based on Hermia’s approach for constant pressure dead-end filtration
laws is proposed to analyze the flux decline behavior during UF in a batch cell. Two
model parameters, namely, complete pore blocking coefficient and cake filtration
coefficient are obtained by minimizing the error involved between calculated and
experimental flux data. These parameters along with known operating conditions,
membrane permeability, and physical properties of feed enable one to predict the
transient permeate flux decline. The effect of various operating conditions, such as
feed solute concentration, stirrer speed, and transmembrane pressure on the flux
decline is studied. Experimental results show that operating conditions have signifi-
cant effect on the onset of cake formation as well as on the flux decline behavior. The
model results are successfully compared with the experimental data.
3.3.1.2 Osmotic Distillation

Osmotic distillation (OD), also called osmotic evaporation or isothermal membrane distillation, can be used to remove water selectively from aqueous solutions under atmospheric pressure and room temperature, avoiding thermal degradation (Courel et al., 2000; Hogan et al., 1998; Kunz et al., 1996). It involves the use of a micro-porous hydrophobic membrane to separate two circulating aqueous solutions at different solute concentrations: a dilute solution and a hypertonic salt solution. If the operating pressure is kept below the capillary penetration pressure of liquid into the pores, the membrane cannot be wetted by the solutions.

The difference in solute concentrations, and consequently in water activity of both solutions, generates, at the vapor–liquid interface, a vapor pressure difference causing a vapor transfer from the dilute solution toward the stripping solution.

Cross-flow UF and OD were implemented on a laboratory scale to obtain formulations of interest for food and/or the pharmaceutical industry starting from the blood orange juice produced in the Calabria region (Destani et al., 2013). Freshly squeezed juice, after a depectinization step, was submitted to an UF process in order to recover natural antioxidants, such as hydroxycinnamic acids, hydroxybenzoic acids, flavanones, flavan-3-ols, and anthocyanins. The UF permeate, with an initial total soluble solids (TSS) content of 10.5°Brix, was concentrated by OD up to a final concentration of 61.4°Brix.

The performance of both processes was analyzed in terms of productivity (permeate fluxes in UF and evaporation fluxes in OD) and quality of clarified and concentrated samples through the identification and quantization of phenolic compounds.

The UF membrane showed a rejection toward the identified phenolic compounds in the range 0.4–6.9% and a little decrease of the TAA (8.2%) was observed in the UF permeate in comparison with fresh juice. Phenolic compounds were also well preserved in the retentate of the OD process as demonstrated by the constant value of the ratio between the concentration of phenolic compounds in the OD retentate and the concentration of these compounds in the UF permeate stream (in the range 5.54–6.39).

3.3.1.2.1 Membrane Fouling and Cleaning

The common practice of membrane cleaning often involves a combination of hydraulic and chemical cleaning. The selection of chemical cleaning agents is largely limited by the compatibility of membrane media and other filter components to cleaning chemicals, and the avoidance of potential product contamination. According to Kuzmenko et al. (2005) and Rabiller-Baudry et al. (2006), membrane cleaning is typically performed daily for 2–3 h after 6–8 h of filtration in the dairy and water treatment industries.

The cleaning agents commonly used for membrane plants are alkalis, acids, enzymes, surface-active agents, formulated cleaning agents, combined cleaning and disinfecting agents, and disinfectants (Ghosh, 2003; Kazemimoghadam and Mohammadi, 2007).

Cleaning membranes fouled with protein solutions is a common part of the normal operational procedure in membrane applications for the dairy and other food
Chemical cleaning with simple caustic and acid cleaners is still a common practice, although the response of different protein components in a foulant layer to cleaning agents is not well understood. Norazman et al. (2013) examined the efficiency of chemical cleaning of PES membranes fouled during UF of whey protein isolate solution and the protein residuals remaining on the membranes after cleaning. Using a combination of the Lowry assay and gel electrophoresis for the estimation of protein amount and composition on the membrane before and after cleaning, it was observed that the high-molecular-weight components in the foulant layer could more easily be removed than the smaller components, and the proteins trapped in the membrane pores were the most difficult to clean. In repeated cycles of filtration and cleaning with NaOH followed by HCl, the flux recovery due to NaOH cleaning remained constant after the second cycle, while the flux recovery due to HCl cleaning increased with the repeated cycle, indicating the importance of HCl cleaning stage in the removal of residual protein components.

The resistance-in-series model was used by Baklouti et al. (2013) to analyze flux behavior, which involved the resistances of the membrane itself, and the fouling and solute concentration polarization. Response surface methodology was used to establish the relationships between operating parameters and UF efficiency and, thus, determined the optimal conditions. Experiments were performed according to Box–Behnken design by changing the levels of three parameters, namely, transmembrane pressure, feed flow rate, and temperature. The fitted mathematical models were employed to plot isoresponse curves. It was shown that the resistance due to solute concentration polarization ($R_{cp}$) dominated the flux decline (40–74%). The fouling resistance ($R_f$) varied from 12% to 46%. To simultaneously optimize the three responses studied ($R_f$, $R_{cp}$, and permeate limit flux), the desirability function approach was applied, which determined the best acceptable compromise.

The selected UF conditions of the compromise were as follows: 3 bars, 0.95 L min$^{-1}$, and 30°C. Optimal values of $R_f$, $R_{cp}$, and permeate limit flux were equal to 18%, 72%, and 19 L h$^{-1}$ m$^{-2}$, respectively.

Many techniques have been implemented to reduce membrane fouling such as hydrodynamic factors considering feed pretreatment, working at critical flux, backwashing, increase in shear at the membrane surface, and use of effective chemical cleaning agents. However, the critical flux approach has opened up interesting perspectives, particularly subcritical flux operations or close to them (Bacchin et al., 2006; Le Clech et al., 2003). The critical flux is a method to minimize fouling and extend the industrial process as much as possible before cleaning steps are necessary. Working at main process parameters (transmembrane pressure and linear flow velocity, largely) near to the critical point, fouling phenomena are drastically reduced and, as a consequence, the productivity and membrane life are significantly increased (Bacchin et al., 1995, 2006; Badan et al., 2008; Field et al. 1995; Iaquinta et al., 2009; Lipnizki, 2008; Metsamuuronen and Nystrom, 2005; Mizubuti et al., 2000; Pollice et al., 2005; Ribeira et al., 2002; Sogi et al., 2003; Wani et al., 2008).

Two UF membranes with different geometries (spiral polymeric and tubular ceramic) but similar cut-offs were used by Muro et al. (2013) to treat wastewater from the food industry. Hydrodynamic conditions were optimized by statistical methods as a strategy to get more accurate values of the critical parameters thereby producing.
and minimizing membrane fouling. The validation of the optimization method was obtained by determining experimental critical flux at critical parameters. Membrane fluxes revealed significant differences during filtration.

The polymeric membrane showed an optimal flux of $45.60 \text{ L h}^{-1} \text{ m}^{-2}$ at 3.21 bar while operating at a stable time of 11.61 h, whereas optimal flux of the ceramic membrane was $32.43 \text{ L h}^{-1} \text{ m}^{-2}$ at 3.98 bar for 16.03 h. Experimental critical flux values were only slightly lower than optimal fluxes for both membranes, showing the validity of the statistics models applied. Negligible osmotic pressure was found on two membranes at critical flux parameters, indicating irreversible fouling for both cases. The polymeric membrane revealed strong fouling behavior and the ceramic membrane showed a weak form; the flux decline occurred first in the polymeric membrane, whereas the ceramic membrane exhibited high stability during the filtration operations. A high degree of purification of wastewater was obtained by this membrane at critical flux conditions.

Cleaning-in-place (CIP) protocols are often automated, requiring that the temperature and chemical composition of the cleaning solution be tailored to the specific foulant. With growing pressure to reduce both the water footprint and environmental impact of a process, CIP procedures must be optimized to minimize water and chemical use, an exercise that will help to reduce costs and cleaning outages. MF and UF are filtration technologies that can separate micron-sized bacteria or even large macromolecules from process streams using a selectively permeable membrane, often without any required heating or energy-intensive mechanical action. Biofilms are associations of microorganisms in an aquatic environment, bound together by an extracellular polymer matrix, and attached as a layer to a substrate such as a pipe or wall. Biofilms are readily deformable soft deposits, which make the use of probes for measurement unsuitable. Researchers have been able to observe their removal from a surface by fluid shear and estimate their initial thickness (Lewis et al., 2012).

Stevioside is one of the naturally occurring sweeteners that can be widely applied in food, drinks, medicine, and daily chemicals. Membrane separation has a potential application in the clarification of stevioside from pretreated stevia extract by UF. Mondal et al. (2013) have used 5-, 10-, 30-, and 100-kDa MWCO membranes. Quantification of membrane fouling during UF is essential for improving the efficiency of such filtration systems. A systematic analysis was carried out to identify the prevailing mechanism of membrane fouling using a batch unstirred filtration cell. It was observed that the flux decline phenomenon was governed by cake filtration in almost all the membranes. For 100 kDa membrane, both internal pore blocking and cake filtration are equally important. Resistance in series analysis shows that the cake resistance is several orders of magnitude higher than the membrane resistance. The cake resistance is almost independent of transmembrane pressure drop, which indicates the incompressible nature of the cake. A response surface analysis was carried out to quantify the development of cake resistance with time during UF of various membranes. Quality parameters show that the 30-kDa membrane is better suited for clarification purposes. Identification of the fouling mechanism would aid in the process of design and scaling up of such clarification setup in future.
3.3.1.2.2 Reverse Osmosis

Osmotic pressure is a critically important property in RO based upon Gibbs free energy, which, on a molar basis, is called the thermodynamic or chemical potential ($\mu$). It is an intensive quantity, that is, dependent on its nature and concentration, but independent of the size of the system.

It is a driving force that describes changes in free energy ($G$) when 1 mol of a component is added to or removed from the system.

This difference in the chemical potential of the water is the driving force for permeation of water from the high-potential side to the low-potential side, a phenomenon called osmosis (Figure 3.4).

The most important structural properties of an RO membrane are its chemical nature, its pore statistics (pore size, pore size distribution and density, and void volume) and its degree of asymmetry. From a functional point of view, the most important are its permeability (measure of the rate at which a given molecule permeates) and its permselectivity (measure of the rate of permeation of one molecule relative to another). These characteristics are more commonly termed as “flux” and “rejection” (Cheryan, 1998) (Figure 3.5).

Selenium-enriched mushroom aqueous enzymatic extracts (MAEE) were obtained from the white button mushroom (*Agaricus bisporus*) by a procedure based on enzyme and membrane technology (Cremades et al., 2012).

The mushroom hydrolysate (MH) was concentrated by three different procedures: vacuum evaporation, RO, and NF at 1 kDa, according to standard procedures.

RO concentration was performed using an RO tubular module (Paterson Candy International, England), which consisted of five perforated stainless-steel tubes connected in series. Each tube was lined with a membrane element, 1.2 m in length and 12.5 mm in diameter (total area of 0.25 m²). The module contained an AFC 80 polyamide (PA) tubular membrane.

![Figure 3.4 Osmosis](image-url)
NF experiments were performed out with tubular membranes (SeIRO MPT-34, Koch Membrane Systems Inc., Stafford, UK) with a 0.5 kDa cut-off. The filtration area was 0.366 m$^2$. Experiments were performed in batch mode, using a laboratory-scale plant.

Concentrations of hydrolysate were of 23.5, 18.2, and 17.3, respectively. Selenium recoveries measured were 71.7%, 65.3%, and 60.1%, respectively. The lower yields observed in the concentration by RO and nF could be attributed to the loss of Se products during the membrane treatments.

MAEE, with a selenium concentration of 51.8 ± 5.58 μg g$^{-1}$, is a product suitable for achieving the recommended daily dose (RDD) of 55 μg with a small amount of around 1 g of product, which can be incorporated into any type of solid or liquid food without modifying its organoleptic properties. Chemical characterization and selenium speciation are also reported; more than 86% of the selenium-containing products are organic in nature. The utilization of this product would help in the treatment and/or prevention of diseases associated with low selenium concentrations, such as aging and neurodegenerative, cardiovascular, and immunological diseases, while avoiding the risk of reaching high plasma selenium concentrations, which has recently been associated with deleterious effects.

RO is a separation method of water from its solutions with the use of membranes having very small diameter pores. It is an alternative to concentration for water removal, but with lower energy consumption. However, it is expensive to buy and maintain, whereas its efficiency depends on the operation conditions (Walstra et al., 1999).

RO aims to concentrate skimmed milk, whey, and liquid waste pollutants with a high microbial load with the final aim their concentration. Operating at low temperatures and the hold-up of volatile substances are some of its advantages whereas the disadvantages are that milk cannot be concentrated at high levels and the filtrate is pure water (Walstra et al., 1999). RO membranes retain particles of MW until 100 Da and values of applied pressure are 5–10 times higher than those of ultrafiltration.
Centrifugation–Filtration

(Rosenberg, 1995). RO of milk and whey removes only water and is similar to thermal evaporation (Mistry and Maubois, 1992). RO is used in whey during powder preparation. Concentration of whey occurs until solids reach 25% followed by evaporation until 50% and then drying. Moreover, RO is applied in whey concentration before ED and in the filtrate arising from UF (Tamime and Robinson, 1999).

RO can be used in milk concentration as well as yogurt preparation. However, it cannot be used in the preparation of strained yogurt since it causes problems due to the increased percentage of lactose and salts in the final product (Tamime and Robinson, 1999).

3.3.1.2.3 Cellulose Acetate Membranes

CA polymers are produced by acetylation of cellulose with acetic anhydride, acetic acid, and a catalyst such as sulfuric acid. Acetylation is carried out until modification of the three hydroxyl groups of each unit occurs (esterified by acetyl groups).

The hydrophilic properties of the membrane are a function of the number of hydroxyl groups remaining after acetylation. Thus, the degree of substitution (DS) must be carefully controlled. The hydrophobic acetyl groups act as cross-links due to dipole–dipole interaction and restrict the swelling resulting in the increased permselectivity of the membrane, that is, water and not hydrated ions are allowed to pass the polymer micellar matrix (Kesting, 1985).

CA membranes are fairly easy to manufacture, and a wide variety of pore sizes are available, from RO to MF. In addition, cellulose (the raw material) is an abundant and renewable resource. There are, however, several limitations to CA that restrict its use, especially in food and biotechnology applications where standards and requirements are quite rigorous. The factors to account for include temperature and pH of operation, use of acidic or basic cleaners, chlorine and other oxidizing agents used for sanitation, microbiological activity, and other mechanical influences such as pressure and high shear in the system.

CA is very temperature sensitive, which limits the maximum operating temperature to 30°C, with some blends of CA and cellulose triacetate (CTA) tolerating 35°C. This low temperature poses several problems such as low flux, since viscosity, diffusivity, and solubility are aided by high temperatures; microbial growth may be a problem at these temperatures (which RO and NF will only make worse since all solutes are concentrated during the process, unless the process is operated at refrigeration temperatures), and cleaning is also more difficult since cleaning agents work best at higher temperatures.

CA is also pH sensitive; recommended pH limits are pH 2–8, preferably pH 3–6. CA will hydrolyze in water at either high or low pH. The rate of hydrolysis, which is lowest at pH 4.5–5.0, is temperature dependent with the recommended pH range decreasing with increasing temperature (Cheryan, 2007).

Thus, the use of MF in the first step of separation can be more effective in subsequent downstream purification by ultra- or NF membrane.

It has been well established that CA polymer can impart high hydrophilicity to a membrane, thus reducing the fouling potential (Kim and Lee, 1998; Muthusamy et al., 2006; Shin et al., 2005; Sossna et al., 2007; Wang et al., 2005, 2006) while the presence of PS can substantially improve mechanical strength. PVP and polyethylene
glycol (PEG) are best-known examples (Wienk et al., 1996) of hydrophilic polymer ingredients that can enhance pore formation on a polymer membrane.

Sikder et al. (2009) focused on synthesis and characterization of a polymer blend MF membrane for separation of microbial cells from lactic acid fermentation broth in a continuous process. The membranes were prepared by blending hydrophilic cellulose diacetate (CA) polymer with hydrophobic polysulfone (PSF) polymer in the wet phase inversion method. Immersion precipitation is the most important and best-studied method in the preparation of phase inversion polymer membranes.

Polymers were blended in N-methyl-2-pyrrolidone (NMP) solvent (70 wt.%), where PEG was added as a pore former. The membranes were characterized in terms of morphology, porosity, flux, and microbial separation capability. The best prepared membrane with PSF/CA weight ratio of 25/75 yielded a pure water flux of 1830 LMH (L m\(^{-2}\) h\(^{-1}\)) and a fermentation broth flux of 1430 LMH at around 1.5 bar TMP (transmembrane pressure). The membrane was successful in the complete retention of microbial cells from the broth in a continuous cross-flow membrane module integrated with the fermentor.

### 3.3.1.2.4 Thin-Film Composites

Hydrophilic polymers are superior to CA. Membranes made of PAs, polyamidehydrazides, polybenz-imidazole, and others are very promising examples (Cheryan, 1998). The mechanical, chemical, and biological properties of PA membranes are generally superior to CA. These membranes are not as susceptible to hydrolysis or to microbial attack and they can tolerate alkaline conditions and temperatures up to 50°C, but are extremely sensitive to chlorine. In the latter respect, they are much worse than CA, tolerating a maximum exposure of 0.1 ppm active chlorine.

The first commercially successful composite membrane is the Dow-FilmTec FT-30 membrane. It is composed of a PS support cross-linked by interfacial polycondensation with the PA polymer. The skin layer may itself be composed of several layers, which enhances its strength, flexibility, and abrasion resistance. The hydrophilic carboxyl groups are responsible for its relatively high water permeability, similar to the hydroxyl groups in CA membranes.

Thin-film composite membranes are available from almost every company that manufactures RO membranes.

For the food industry, composites are available as spirals, as flat sheets in plate systems, and in tubular form. It has been used in seawater desalination and has been field tested in food processing facilities for several years (Cheryan, 2007).

Composite hydrophilic pervaporation membranes were prepared from chitosan (CS) blended with hydroxyethylcellulose (HEC) using CA as a porous support by Jiraratananon (2002). The membrane with a CS/HEC blend ratio of 3/1 exhibited the highest pervaporation separation index (PSI) and was selected to be cast on a porous CA support.

### 3.3.1.2.5 Nanofiltration

NF is effective in separating the mixtures of small organic solutes such as oligosaccharides, low-MW peptides, inorganic salts, amino acids, and other low-MW materials.
NF, which is widely used in many industrial sectors, offers several advantages such as low operating pressure, high flux, and high retention of multivalent anion salts and organic molecules (Eriksson, 1988). In addition to the capability of reducing the ionic strength of the solution, NF membranes can remove hardness ions, organics, and particulate contaminants. NF has been employed in water treatment (Bowler et al., 1998; Frenzel et al., 2006), the food industry (Samhaber, 2005), pretreatment for desalination (Hassan et al., 1998), and other areas.

The use of a porous microresonator placed in a microelectrofluidic system for integrated functions of NF and sensing of small biomolecules and chemical analytes in extremely dilute solution was proposed and investigated by Huang and Guo (2012). As an example, aminoglycosides in drug residues in food and livestock products were considered as the trace chemical analyte. The filtration process of the charged analyte in aqueous solution driven by an applied electrical field and the accompanying optical whispering-gallery modes in the resonator are modeled. The dynamic process of adsorption and desorption of the analyte onto the porous matrix is studied. Deposition of the analyte inside the porous structure will alter the material refractive index of the resonator, and thus induce an optical resonance frequency shift. By measuring the optical frequency shift, the analyte concentration as well as the absorption/desorption process can be analyzed. Through an intensive numerical study, a correlation between the frequency shift, the analyte concentration, and the applied electrical voltage gradient was obtained.

This reveals a linear relationship between the resonance frequency shift and the analyte concentration. The applied electrical voltage substantially enhances the filtration capability and the magnitude of the optical frequency shift, pushing the porous resonator-based sensor to function at the extremely dilute picomolar concentration level for small bio/chemical molecules down to the subnanometer scale. Moreover, the use of the second-order whispering-gallery mode is found to provide better sensitivity compared with the first-order mode.

NF separates mixtures of proteins and peptides of MW from 300 to 3000 Da. NF can concentrate organic compounds with the removal of monovalent ions such as sodium and chlorine with the result the desalination (Kilara, 2006). Some NF membranes can be used for desalination when high pressures are applied and this is an alternative separation method for ED (Walstra et al., 1999).

NF membranes are membranes where pressure is applied targeting on the small retention of monovalent ions (Kelly et al., 1992). Separation characteristics of NF fall between RO and UF (Jelen, 1992). Applied pressures range between 6 and 40 bar, whereas in RO, pressures are applied until 40 bar, and in UF, they fall below 10 bar (Jeantet et al., 2000). The mechanism of separation of NF membranes depends on the simultaneous effect of electrical separation and separation based on the size of particles. NF membranes do not have visible pores, but have free pores with different structure and opening (Nyström et al., 1995). Most commercial NF membranes are fine synthetic membranes bringing an active layer consisting of aromatic PAs and are negatively affected at neutral pH values (Yaroshchuk et al., 2000).

NF has applications in whey concentration with the retention of high lactose quantities. By using NF, whey is concentrated approximately 15–25% and total inorganic compounds are reduced by 40–50% whereas losses in lactose range between 1% and
5% (Van der Horst et al., 1995, Vasilievic and Jelen, 2000). Moreover, NF applies in the production of fresh whey cheese (Zambrini et al., 1990) and in the recovery of amino acids and peptides from the hydrolysis of β-lactoglobulin (Wijers et al., 1998). NF may be used by the yogurt industry to remove part of lactose from the filtrate of milk UF, which will then be used in yogurt manufacturing, because the product will have a lower percentage of lactose making it more digestible (Rinaldoni et al., 2009).

A dynamic mathematical model was developed by Dey et al. (2012) for a flat sheet cross-flow NF membrane module for the separation of lactic acid from fermentation broth. The model developed with extended Nernst–Planck approach included pH effects during Donnan exclusion of lactate ions through the Henderson–Hasselbalch equation.

The most relevant structural characteristics (pore radius, porosity-to-thickness ratio, and membrane charge density) of the membranes were completely determined by comparison and convergence of the model-predicted and experimental data on flux and rejection using standard solute–solvent systems as well as actual fermentation broth. Thus, the pore sizes of the investigated three NF membranes NF2, NF3, and NF20 were determined as 0.57, 0.55, and 0.54 nm, respectively. Through the modeling and experimental investigation, the best membrane (NF2) that could retain and recycle more than 90% sugars while allowing more than 70% lactic acid to permeate could also be selected. Finally, the refined model could very well predict transport of lactic acid through NF membranes operating in flat-sheet cross-flow module as evident in a very small relative error (<0.1) and the value of overall correlation coefficient ($R^2$) of greater than 0.980.

Economic evaluation of a membrane-integrated bioreactor system for lactic-acid production from sugarcane juice was performed by Sikder et al. (2012). The production process consisted of sterilization, fermentation, MF, NF, and final concentration by vacuum evaporation. Membrane recycle fermentor operating at a cell concentration of 22 g L$^{-1}$ resulted in a productivity of 53 g L$^{-1}$ h$^{-1}$ with a lactic acid concentration of 106 g L$^{-1}$ and a yield of 0.96. The membrane units (cross-flow MF and NF) and pump contribute about 2% to the total fixed capital cost whereas fermentation unit along with holding tank contribute about 36% to the total fixed capital cost. The two largest cost components were raw material and yeast extract costs contributing about 6% and 87%, respectively, to the total operating cost. Total product cost stood at 3.15 US$ kg$^{-1}$ of 80% (w/w) concentrated and 95% pure lactic acid. The study revealed that the operating cost could be reduced further by using a cheaper nitrogen source such as silkworm larvae or yeast autolysate and installing the lactic acid plant in the sugarcane-growing areas or by optimizing the recycle of NF retentate to the fermentor.

Olive milling produces huge amounts of wastewater (OMWW) characterized by an extremely high organic load. Its polyphenols content is a hindrance to conventional biological treatment and as a growing medium for common microbial bio- masses. The practice to dump it on soil is in conflict with the latest EU directives on waste management.

OMWW can be effectively and efficiently treated by means of membrane technology to a fraction of the initial volume, but membrane-processing concentrates still require treatment. Reversing the overall cost balance of membrane processing and subsequent treatment requires valorizing the concentrates through their reuse,
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as well as ensuring long-term service of the membrane system through effective wastewater pretreatment and sustainable, fouling-controlling, membrane operation conduits.

Cicci et al. (2013) reused and valorized the ultra- and NF-membrane concentrates as media for biomass production of microalgae and cyanobacteria. *Scenedesmus dimorphus* and *Arthrospira platensis*, usable as a food, feed, nutraceutical component, or feedstock for biofuels, were selected for this investigation. The performances on microalgal growth obtained by using the different membrane concentrates and the pretreated olive mill wastewater (OMWW) feedstock were experimentally determined and related to the composition of the culture media thus obtained. They were also related to the irradiance distribution within the photobioreactor volume to decouple light limitation and medium chemical composition effects in the production of microalgal and cyanobacterial biomass.

Climate changes are inducing increased sugar levels of must, which produces negative effects on wine quality as unbalanced wines with high degrees of alcohol. So, effective strategies to control the increase of sugar levels in must have been studied. The most recently applied technologies are based on the use of membrane processes that retain the sugars of musts (Bonnet and De Vilmorin, 2004; Calvin, 2001; García-Martín et al., 2009; Gresch, 1996).

One of them is the use of a membrane process, and this is applied in the work by Mihnea et al. (2012). Single NF treatments to the initial must were applied. Three different types of membranes, HL (HL2540FM), DL (DL1812C-28D), and DK (DK1812C-28D), all provided by GE Water & Process Technologies (Barcelona, Spain), were used. According to the commercial company, DL is characterized by 96% cut-off retention for magnesium sulfate and 2.27 water permeability, and DK by 98% cut-off retention for magnesium sulfate and 1.98 water permeability. A combination of UF and NF was also studied.

The sugar level of white must from Verdejo (*Vitis vinifera* variety) was reduced using diverse membrane processes, and the effect of this on the volatile composition of the corresponding wines is studied. The study was carried out during three consecutive vintages. An important impact of the reduction of sugar levels of must on the volatile composition of the obtained wines was detected, which was due to some retention phenomena of aromatic and precursor compounds. To minimize the volatile composition modifications, an appropriate selection of the NF membrane must be done.

During desalination of feed with highly concentrated salt by NF, predictive modeling was difficult due to the effect of salt on the retention of organic solutes. Consequently, a better understanding of the salt effect on membrane and organic solutes was required. Luo and Wan (2011) used four well-known commercially available NF polymeric membranes, NF270, NF-, Desal-5 DL, and Nanomax50, and analyzed them by a model based on an extended Nernst–Planck equation, using highly concentrated glucose and sodium chloride (NaCl) solutions. The results showed that with increasing salt concentration, the solute-to-pore size ratio ($\lambda_i$) decreased while the ratio of effective membrane thickness to porosity ($\Delta x/\varepsilon$) increased, indicating that the effect of salt may include decreasing solutes size, increasing membrane pore size, and increasing effective membrane thickness. Moreover, such salt effect
appeared to be independent of membrane and solute types, and the correction model could well predict the retention of charged solutes at high salt concentration because electrostatic repulsion effect between charged solutes and membranes was completely screened by the salt ions. Meanwhile, several hypotheses such as membrane swelling, hydration layer thinning, and particle collision were provided to explain the change of model parameters by highly concentrated salt.

Bouchoux et al. (2005) and Bargeman et al. (2005) proposed that membrane swelling (i.e., an increase of average pore size) occurred due to the stronger repulsive interaction between ions with same charge inside the pores when salt was added, implying that electrostatic effect was enhanced by increasing salt concentration. However, such explanation contradicted charge screening by salt. Nilsson et al. (2008) argued that membrane swelling due to a salting-in effect (also called lyotropic effect (Piculell and Nilsson, 1990)) was more suitable to explain their experimental results.

It is well known that pH and salt conditions have significant influence on NF performance. In order to manipulate NF process and optimize its efficiency, it is very important to get insights into the effects of pH and salt on NF. Luo and Wan (2013) reviewed the reports on NF performance at different pH and salt conditions, focusing on the mechanisms behind various phenomena induced by pH and salt. The effects of pH and salt on NF are mainly reflected in the variations of membrane flux/permeability, solute rejection, and fouling behavior, which also depend on both solute type and solution composition. In order to explain these effects, the changes of membrane properties are evaluated by physical, chemical, and mathematical characterization methods. Eight mechanisms for pH and salt effects are summarized and several practical advices for NF operation are provided. Besides, some interesting opinions such as dominant ions, co-ions competition, salting-out induced pore swelling, and charge-induced concentration polarization are reviewed. This review also intends to provide a guide to optimize NF separation and maintenance.

3.3.1.2.6 Diafiltration
DF is a useful membrane filtration technique to separate macrosolutes from microsolutes based on their molecular size difference (Paulen et al., 2011). In view of its advantage in the maximization of the purity of desired substances such as proteins, polysaccharides, and so on, the DF process combined with a UF or NF membrane was widely studied. For instance, Gonzalez-Munoz and Paraja (2010) operated the aqueous eucalyptus wood DF through a ceramic NF membrane, and developed a mathematical model to predict the time-course of flux (Jv) and the concentrations of the different compounds. The retention was also introduced, which was considered as a constant. Sun et al. (2011) demonstrated the potential application of UF for the recovery of the polysaccharides from rapeseed, as a key step in the large-scale development of rapeseed meal. The MW distribution of the polysaccharides in bunk solution or the solutions rejected by the membranes was analyzed by size exclusion chromatography. However, the MW distribution of the polysaccharides in the permeate was not determined, and the retention of each MW fraction of polysaccharides was not analyzed during DF.

The polysaccharides were considered as total carbohydrates, and the recovery and purity were computed by a calibration curve of absorbance at 530 nm.
To investigate the membrane separation performance of multicomponent solution throughout continuous volumetric diafiltration (CVD), sucrose and glucose were implemented as model solute (Zhao et al., 2013). Two commercial NF membranes (QY-NF-1-SW with MWCO of 150 Da and QY-NF-3-D with MWCO of 250 Da) were operated in batch recycling mode. Flux ($J_v$), transmembrane pressure ($\Delta P$), concentrations in feed ($C_f$), and permeate ($C_p$) were collected, and the retentions ($R$) were calculated. Based on the Spiegler and Kedem (S–K) equation, a theoretical $R$ model was established. The model was employed to establish the mass balance equations in a feed tank during CVD. A novel revised retention equation was established to simulate CVD of sucrose and glucose mixture solution. Further, the model was popularly applied in UF (UF) of the multicomponent solution—soybean molasses by a commercial membrane (QY-UF-1-G with MWCO of 1 kDa). It was predicted in theory that the total retention of multicomponents increased, since the concentration of low-retention solute declined with DF process time when the flux was insignificantly changed, which was then demonstrated by the experiment. The results showed that the retention curve predicted by the model agreed well with the experiment, and the revised retention equation was well designed for industrial food fluids.

3.3.1.2.7 Microfiltration

Carrere et al. (2002) dealt with the first unit operation of the downstream process for the production of lactic acid: the clarification of fermentation broths by cross-flow MF. MF experiments conducted under constant transmembrane pressure and under constant permeate fluxes (higher and lower than the critical flux) were represented by the resistance in series model in which the membrane resistance, the adsorption resistance, the bacteria cake resistance, and the soluble compounds concentration polarization resistance were taken into account. The different operating modes were compared in terms of two industrial interest criteria: the productivity and fouling rates. Higher productivities were obtained during constant transmembrane pressure runs whereas the lowest fouling rate was observed during the run conducted with a constant permeate flux lower than the critical flux. However, this fouling was mainly due to adsorption and solute components concentration polarization.

Concentration polarization refers to the reversible accumulation of solute within a thin boundary layer adjacent to the membrane surface (Blatt et al., 1970).

The process for the manufacture of milk protein concentrate (MPC) powders involves the UF and DF, and optional evaporation of the retentate prior to drying. MPC powders may be made as low- or high-heat products (Getler et al. 1997; Huffman and Harper, 1999). MPC powders have been used in cheese and a range of food applications, including meat, bakery and dairy products, high protein drinks, and desserts (Zwijgers, 1992).

The solubility of MPCs powders was influenced by the method used for preparing the concentrate, drying conditions, and the type of dryer used (Augustin et al., 2012). Increasing total solids of the ultrafiltered concentrates (23% total solids, TS) by DF to 25% TS or evaporation to 31% TS decreased the solubility of MPC powders (80–83% protein, w/w dry basis), when UF was followed by evaporation a higher total solids was obtained and this had the greater detrimental effect on solubility. High shear treatment (homogenization at 350/100 bar, microfluidization at 800 bar
or ultrasonication at 24 kHz, 600 W) of ultrafiltered and diafiltered MPCs prior to spray drying increased the nitrogen solubility of MPC powders (82% protein, w/w dry basis). Of the treatments applied, microfluidization was the most effective for increasing nitrogen solubility of MPC powders after manufacture and during storage. Manufacture of MPC powders (91% protein, w/w dry basis) prepared on two different pilot-scale dryers (single stage or two stage) from MPCs (20% TS) resulted in powders with different nitrogen solubility and an altered response to the effects of microfluidization. Microfluidization (400, 800, and 1200 bar) of the concentrate prior to drying resulted in increased long-term solubility of MPC powders that were prepared on a single-stage dryer but not those produced on a two-stage spray dryer. This work by Augustin et al. (2012) demonstrates that microfluidization can be used as a physical intervention for improving MPC powder solubility. Interactions between the method of preparation and treatment of concentrate prior to drying, the drying conditions, and dryer type all influence MPC solubility characteristics.

A novel method for the extraction of (−) epigallocatechin gallate of high purity from green tea leaves is proposed by Kumar et al. (2012). The method comprised a two-stage water-based extraction followed by successive use of MF and UF. MF was used as a pretreatment to UF. The best process conditions of each unit operation were estimated by performing well-planned experiments. The clarified liquor was dried to powder by freeze drying. Chemical analyses revealed that the tea powder contained about 90% of polyphenols. The purity of (−) epigallocatechin gallate was found to be about 80%, while its average yield was 1.22 g L\(^{-1}\). The method outlined in this study may have remarkable importance for the bulk production of high-purity (−) epigallocatechin gallate with potential applications in pharmaceutical, cosmetic, and food processing industries.

Besides being a green process, this method can be easily scaled up for the commercial production of (−) epigallocatechin gallate.

MF removes small-size particles such as bacteria, yeast cells, and colloid particles. The pores of the MF membranes have a size ranging between 0.1 and 10 \(\mu\)m and retain and separate the above-mentioned particles (Huisman, 2000). Pressures applied are lower than those applied in UF membranes, ranging between 0.1 and 8 bar, with a higher flow (Rosenberg, 1995).

Hemicelluloses with a high molecular mass are needed for the manufacture of value-added products such as food packaging barrier films. Krawczyk et al. (2013) recovered such molecules from chemithermomechanical pulp (CTMP) process water using an innovative three-stage process comprising membrane separation and enzymatic treatment with laccase. MF followed by UF was found to be a suitable combination in the first stage, providing a concentrated and purified hemicellulose fraction suitable for enzymatic treatment.

The MF membrane was a tubular ceramic membrane manufactured by Atech Innovations GmbH (Gladbeck, Germany). This membrane had seven parallel feed channels, each with an inner diameter of 6 mm. The total length of the membrane tube was 1 m and the outer diameter was 25 mm. Spiral-wound elements (Alfa Laval Nordic A/S, Soborg, Denmark) equipped with a 48-mil spacer were used in both UF stages. The UF membranes were made of the same material, but differed in their nominal MWCO.
In both membrane processes, a high average flux (260 and 115 L m$^{-2}$ h$^{-1}$) and a low fouling tendency were observed. A marked increase in the average molecular mass of hemicelluloses with bound lignin moieties was achieved by laccase treatment in the second stage. The enzymatically cross-linked hemicelluloses were finally recovered in the third stage using UF. In the final high-molecular-mass solution, the hemicellulose concentration was 54 g L$^{-1}$, the contribution of hemicelluloses to the total solids content was 43%, and the viscosity of the solution was 27 mPa s. The results demonstrate that a hemicellulose fraction of high quality can be produced from CTMP process water, and that this could constitute a suitable feedstock for the production of, for example, barrier films for renewable packaging.

MF was initially developed in Germany in 1929 from Sartorius-Werke. During the Second World War it was developed for bacteriological examination of water supplies. Until 1963, the manufacturing material of MF membranes was nitrocellulose or a mixture of cellulose esters (Merin and Daufin, 1990). Nowadays, these membranes are manufactured by glass, ceramic materials such as alumina, titanium dioxide, and zirconium oxide (Figure 3.6), and metals such as silver and stainless steel. The advantage of these inorganic compounds is their stability over extreme conditions during food processing, such as high temperature values, extreme pH values, and the contact with solutions different in composition than water. According to Espina et al. (2010), ceramic membranes in MF have a better yield compared to organic membranes during casein micellar separation from whey proteins in skimmed milk. Most metallic and some ceramic membranes are manufactured with concentration of their materials without melting, whereas the remaining ceramic membranes are manufactured with the fusion of a liquid colloidal system or with anode oxidation. Some new membranes are prepared with lithographic techniques (Espina, 2010).

In 2002, MF market showed profits of approximately 400 million dollars and the annual growth rate was 6.6% (Huisman, 2000).

MF can take place in two different ways: (a) dead-end filtration, in-line filtration during which the direction of flow is perpendicular to the membrane and (b) cross-flow or tangential MF, where the direction of flow is tangential to the membrane.
During dead-end MF, particles continuously move toward the direction of membrane and are deposited on its surface or within the pores of the membrane. This deposition leads to an increasing resistance to flow with the result of the continuous reduction in the rate of filtrate flow. The reduction in the deposited precipitate is accomplished by the application of tangential MF. The tangential flow to the membrane removes particles from the membrane surface and minimizes the particle deposition (Huisman, 2000).

MF is used for the manufacture of milk, cheeses, and products with a high shelf life such as milk powder and milk proteins because it reduces the bacteria population and increases their shelf life. In yogurt manufacturing, MF is used in conjunction with UF. When it is used before UF, it contributes to the maintenance of the membrane pores of UF, that is, keeping them clean and minimizing the deposition of particles on the membrane, keeping the flow at high rates. Moreover, it can be used in whey powder manufacturing since it can retain liposomes of small diameter that have escaped centrifugation (Rinaldoni et al., 2009) and can contribute to the increase of purity.

### 3.3.1.2.7.1 Application of Microfiltration in the Dairy Industry

The diameter of the pores of MF membranes range between 10 and 0.1 μm and can be applied in specific particle separations dispersed in liquids. Milk particles can be separated according to their size as follows: somatic cells (15–6 μm), liposomes (6–0.2 μm), bacteria (15–0.2 μm), and casein micelles (0.3–0.03 μm) (Pierre et al., 1998).

Milk MF should be carried out with special attention to avoid the quick blocking of the membrane pores. Initially, the device should be cleaned with hot water (52°C) and with evaporation valves open in order to remove air bubbles. Then, the heating of the milk takes place at 50°C for 20 min with the aim to assure the physicochemical balance of milk (Saboya and Maubois, 2000).

An alternative technique to that of MF of milk is bactofugation. With this technique, specially designed separators centrifuge milk at 73°C, so bacteria being a bit more heavier than milk are removed with a small quantity of skimmed milk. With bactofugation, we also have removal of spores in the milk (Walstra et al., 1999). This technique requires high amounts of energy and the reduction in the total number of spores ranges between 90% and 95% (Guerra et al., 1998).

Microfiltrated milk contains approximately 3.5 log microbial load lower than conventional milk. Moreover, sporogenic bacteria surviving pasteurization can be better retained with MF due to the large cellular volume. The number of spores using MF decreases by 4.5 log. Regarding the effect of MF on pathogenic bacteria such as *Listeria monocytogenes*, *Brucella abortus*, *Salmonella typhimurium*, and *Mycobacterium tuberculosis*, their number diminishes by 3.4, 4, 3.5, and 3.7 log, respectively. Hence, microfiltrated skimmed milk contains pathogenic bacteria at a concentration of less than 1 cfu L⁻¹ (Madec et al., 1992). MF also removes somatic cells fully resulting in microfiltrated milk not containing their enzymes resistant to pasteurization (Law and Goodenough 1995).

### 3.3.1.2.7.2 Microfiltration Applications in the Food and Drink Industry

An important application of MF is in fruit juices. Several researchers have reported on
the effect of microfiltration on orange and apple juice as well as tropical fruits. One of the basic organoleptic characteristics of fruit juices is the aroma that depends on the volatile compounds, which in turn depend on thermal processing. Similarly, vitamins are sensitive during thermal processing and the presence of oxygen. Pasteurization at 90°C assures the microbiological safety of the juice; however, it negatively affects its organoleptic characteristics. MF is another alternative of juice processing (Matta et al., 2004). During thermal processing of apple juice, natural volatile aromatic compounds diminish, hence affecting its nutritional value (Perédi et al., 1981). However, using MF, concentrations of aromatic compounds increased except that of iso-butyl-acetic acid, whereas pasteurization of microfiltrated juice reduced their concentrations (Su and Wiley, 1998). Moreover, using MF membranes, yeasts and molds were removed fully in apple juice and its clarity was accomplished with removal of solids (Matta et al., 2004).

MF is also applied in brewing. Clarification of produced beer is important for product stability and is achieved with removal of active yeast cells and molecules formed with the interaction of phenolic and protein particles at low temperatures (colloid stability). Moreover, it is very important to retain aromatic compounds and reduce bound oxygen in beer to keep the aroma constant (Gana et al., 2001). Kieselguhr filtration is the well-known technology of beer filtration. However, the high cost leads to the use of MF membranes. Ceramic membranes with pores of 0.2–1.3 μm can easily remove solid substances and yeasts, reduce losses in beer production, and reduce energy required to manufacture a qualitative product (Kiefer, 1991).

In vinegar and wine production, MF is used as a pretreatment to remove solids and sterilize the product. In wineries, MF (a) stabilizes wine by removal of colloid particles, (b) avoids secondary fermentations with bacteria and yeasts removal and (c) clarifies wine (Van der Horst and Hanemaaijer 1990).

MF also removes ovomucin from egg white. It is a glycoprotein of high MW affecting the viscosity of egg white (Powrie and Nakai, 1986). According to Ferreira et al. (1999), MF membranes with pores of 1.4 μm reduced the number of microorganisms in egg white and separated the glycoprotein without blocking the membrane pores.

Finally, MF is used in the processing of liquid waste. Separation of water from oil emulsions is a very important process. Classical methods include chemical demulsification and precipitation. However, high energy consumption is required at a high cost. Hence, MF can be used alternatively (Hu and Scott, 2007). Many researchers have used MF to reduce pollutants of water-in-oil emulsions. Anderson et al. (1987) effectively separated oil emulsions with the use of MF. Moreover, MF was used as a pretreatment for the further processing of liquid wastes.

Li et al. (2013) used MF membranes to remove solid particles from the liquid waste of a fish processing industry followed by UF.

3.3.1.2.7.3 Cleaning of Microfiltration    Water should not contain any colloid particles or microorganisms that could block the membrane pores. Washing should be done with hot water (50°C) followed by washing with an acid and then with a base. Finally, washing with deionized water takes place until the pH of water is neutral (Beolchini et al., 2005). The efficiency of washing is controlled with water flow and
the bacteriological condition of the last washed water, which should not contain any microorganism (Saboya and Maubois, 2000).

3.4 CERAMIC MEMBRANE ELEMENTS AND MEMBRANE FILTRATION SYSTEMS

GEA Westfalia Separator Group (2013b) develops, manufactures, and installs ceramic membranes and complete membrane filtration systems. Thanks to cross-flow filtration with ceramic elements, a solution is now available from a single source for many application areas requiring the parallel employment of separators, decanters, and membrane filtration.

Benefits to the user: Intelligent technologies using centrifugation and membrane filtration, matched to each other, providing a significant improvement in the process line efficiency, for example, in the following applications:

Food industry
- Production and processing of dairy products
- Clarification of fruit juices, wine, and beer

Clarification, fractionation, and concentration of fruit juices and plant extracts have been described by Todisco et al. (1998), Jiao et al. (2004), Rai et al. (2006a, b), and Sarkar et al. (2008a) employing the use of UF, NF, and RO. However, the disadvantage of RO is related to its inability to reach high concentration levels because of limitations imposed by high osmotic pressures.

Single-stage RO systems permits to reach final concentrations of fruit juices of about 30°Brix (corresponding to osmotic pressures of 50 bar) quite below the value of 45–65°Brix for standard products obtained by evaporation (Rodrigues et al., 2004).

- Water treatment

Biotechnology
- Continuous contamination-free separation of biotechnologically recovered products from fermenters

Chemical industry
- Catalyst recovery
- Purification and recovery of raw materials
- Recycling of solvents

Recycling technology
- Water treatment
- Recycling of cleaning baths
- Oil–water emulsion separation

Different membrane configurations are shown in Figure 3.7.
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Hollow fiber refers to very-small-diameter membranes. The most successful one has an outer diameter of only 93 mm and is used for RO.

Low-pressure and high-pressure hollow-fiber membrane modules exist. For low-pressure applications such as air, they may run with tube-side feed. Gas membranes operating at high pressure (above 1.5 MPa) are almost always run with shell-side feed. The outer diameter for gas membranes may be as high as 500 mm (Perry and Greene, 1984; Perry's, 1999).

The spiral-wound module is used in many membrane processes. Permeate collection material is wound on a perforated permeate pipe. A membrane “sandwich” is

![Diagram of membrane configurations including plate and frame, hollow fiber and spiral cartridge.](image)
constructed over the permeate carrier using glue seams as seals. Membrane “sand-
wiches” are separated by feed-channel spacers, through which the feed stream is
passed (Figure 3.7).

The plate and frame module is very much like a filter press. Once found in RO,
UF, and MF, it is still the only module commonly used in ED.

3.4.1 Technology and Function of the Ceramic Membrane Elements
Dynamic filtration with ceramic membrane elements involve coupling with the low-
est possible flow resistance using a support made of pure $\alpha$-$\text{Al}_2\text{O}_3$ with a macropo-
rous structure. The membrane is applied to this support material and consists of at
least one, but usually several layers of highly porous ceramic, with a precisely defined
texture. The layer with the finest porosity determines the filtration characteristics.

GEA Westfalia Separator Group offers an unrivaled broad, application-specific
spectrum of membranes and element geometries. Membranes with rated pore sizes of
1–1400 nm are available. Ceramic membranes are therefore ideal for use in MF and UF.

The superb properties of such membrane elements are used successfully in filter
systems from GEA Westfalia Separator Group worldwide and have the following
advantages:

• Inert material
• Acid and lye resistant
• Solvent resistant
• Regenerative
• Backflush no problem
• Wear resistant
• Long service life
• Reliable
• Heat resistant, steam sterilizable

Centrifugal technology such as the Westfalia Separator’s FRUPEX process is
commonly used since 2001 by juice manufacturers because it gently separates the
liquid from the solids and can be tightly controlled. The company’s ESE 500 clari-
fier was specially designed for this application and can process up to 50,000 L h$^{-1}$.

The demand for beer and fruit juice in countries such as China and Russia is grow-
ing and offers new opportunities to the beverage industry. The need for high-perfor-
ance production machines parallels this. Recently, Westfalia Separator’s FRUPEX
process, comprising a decanter in the first stage, a decanter in the second stage, and
a clarifier, was installed for the first time in the main apple-growing region in China.

Westfalia Separator has introduced a new process that uses centrifugal technol-
ogy to obtain the “wine must,” that is, juice from the grapes.

Known as the VINEX process, it produces a rapid, gentle, and hygienic preclari-
fication of the wine must. The red wine mash is fed into the rotating decanter bowls,
where pressure forces the grapes against the wall of the bowl and the centrifugal
force separates the must from the solid components. The system continuously dis-
charges the must, which is taken away for fermentation. The red wine mash is heated
Centrifugation–Filtration

The remaining pomace, consisting of grape pips and skin, is conveyed by a scroll out of the bowl. The bitter grape pips are not damaged in the process and can, for example, be pressed to obtain grape pip oil. The remainder of the pomace can be turned into compost (Westfalia, 2001).

3.4.2 Ceramic Multichannel Element

The ceramic multichannel element, consists of highly porous ceramic containing several rounds of channels running parallel to its longitudinal axis on the surface of which the membrane is mounted (Figure 3.8).

The suspension to be clarified flows into the channels along the membrane, whereby a partial stream passes through the membrane as filtrate and is discharged by the carrier material. Owing to the very high permeability, the pressure loss on passing through the carrier is so low that it is negligible compared to the pressure drop when passing through the extremely thin membrane.

3.5 Centrifugal Separations

In a centrifuge, great forces can be obtained by the centrifugal action. Gravity still acts and the net force is a combination of the centrifugal force with gravity as in the cyclone; however, gravity can be easily neglected since its forces are very weak.

Centrifugal force is generated when materials are rotated around an axis. The size of the force depends on the radius and speed of rotation and the density of the centrifuged material (Fellows, 2000). The force generated through the rotation acts in an
outward direction. Depending on the speed of the rotating body, it increases or drops on the circular path. Mechanical separation technology makes use of this property when light substances, heavy substances, or substances of different densities have to be separated from each other.

Separation by means of centrifugal force is, however, faster when the vessel has an insert. The heavier particles deposit faster due to the insert. The settling path is shortened by the insert and hence a higher throughput capacity is attained. This means that larger volumes of liquid mixtures can be clarified or separated in the same period of time. The more inerts there are, the shorter the settling paths and the higher the throughput capacities.

The centrifugal force on a particle that is constrained to rotate in a circular path is given by

\[ F_c = mr\omega^2 \]  

where \( F_c \) is the centrifugal force acting on the particle to maintain it in the circular path, \( r \) is the radius of the path, \( m \) is the mass of the particle, and \( \omega \) the angular velocity of the particle = \( 2\pi N/60 \) (Earle, 1983; Heldman and Hartel 1997a, b).

And since \( \omega = v/r \), where \( v \) is the tangential velocity of the particle

\[ F_c = (mv^2)/r \]  

The centrifugal force depends upon the radius and speed of rotation and upon the mass of the particle, and the heavier the particle, the greater the centrifugal force per unit volume.

A boundary region between the liquids at a given centrifuge speed forms at a radius \( r_n \), where the hydrostatic pressure of the two layers is equal. This is termed the neutral zone and is important in equipment design to determine the position of feed and discharge pipes.

If we have a dense and light liquid, then

\[ r_n = (\rho_d r_d^2 - \rho_l r_l^2)/(\rho_d - \rho_l) \]

where \( d \) and \( l \) represent the dense and light liquid layers, respectively, \( \rho \) is the density in kg m\(^{-3}\), and \( r \) is the radius in m.

If liquid flow is streamlined, the rate of movement and volumetric flow rate is determined by the densities of the particles and liquid, the viscosity of the liquid, and the speed of rotation (Equation 3.2).

\[ Q = \frac{D^2 \omega^3 (\rho_p - \rho_l) V}{18 \mu \ln(r_2/r_1)} \]

where \( Q \) is the volumetric flow rate (m\(^3\) s\(^{-1}\)), \( V \) is the volume of the centrifuge (m\(^3\)), \( D \) is the diameter of the particle (m), \( \rho_p, \rho_l \) are the density of particles and liquid,
respectively, in kg m\(^{-3}\), \(\mu\) is the viscosity of the liquid (N s m\(^{-2}\)), and \(r_2, r_1\) are the radius of centrifuge bowl and radius of liquid, respectively, in m, and \(N\) is the speed of rotation in rev s\(^{-1}\).

The residence time \(t\) is \(= V/Q\), that is, the time taken for a particle to travel through the liquid to the centrifuge wall (Fellows, 2000). Derivations and additional details of these equations are given by Brennan et al. (1990) and Earle (1983).

Secondary flows result from curvature as nonuniform centrifugal force-induced Dean vortices (Dean, 1927). As a result, cross-sectional mixing occurs, decreasing residence time distributions (Saxena and Nigam, 1984) while substantially increasing the heat transfer (Shah and Joshi, 1987). Density differences between the bulk and the wall induce Morton vortices (Morton, 1959), which promote cross-sectional mixing and increase heat transfer (Morcos and Bergles, 1975). When curvature and buoyancy forces are both significant, a mixed flow regime can be observed.

The heating, holding, and cooling section of the high-temperature region (\(T > 100^\circ\text{C}\)) of coiled UHT sterilizers were simulated three-dimensionally by Kelder et al. (2002) to assess the impact of centrifugal and buoyant forces on viscous power-law food products. For a wide range of curvature (De = 0–500), heat transfer, lethality development, and thiamine concentration were analyzed.

Curvature and pseudoplasticity substantially increase heat transfer and render the axial velocity profile more uniform. As a result, processing times in the heater, holder, and cooler can be shorter, thus greatly improving thiamine retention. Significant lethality accumulated in the heating section, but the cooling section contributed very little to product sterility. Buoyant forces were shown to be of minor importance in coiled sterilizer flow at the current process conditions.

Decanoic acid reverse micelle-based coacervates were proposed by Garcia-Prieto et al. (2008) for the extraction of bisphenol A (BPA) from canned vegetables and fruits prior to its determination by liquid chromatography and fluorescence detection at \(\lambda_{\text{exc}} = 276\ \text{nm}\) and \(\lambda_{\text{em}} = 306\ \text{nm}\).

Coacervates are water-immiscible liquids that separate from colloidal solutions under the action of a desolvating agent, affecting the temperature or the pH of the colloidal solution (Gander et al., 2002). The procedure involved the extraction of minute quantities (300–700 mg) of homogenized food sample with an aqueous solution containing 10% of THF and 0.5% of decanoic acid, conditions under which the coacervate (around 340 \(\mu\)L) formed \textit{in situ} and instantaneously. The overall sample treatment, which included extraction and centrifugation, took about 25–30 min, and several samples could be simultaneously treated using conventional lab equipment. No clean-up or solvent evaporation were required. Extraction efficiencies mainly depended on the decanoic acid and THF concentration in the aqueous solution and were not affected by the pH or the temperature in the ranges studied (1–4 and 20–60°C, respectively). Recoveries in samples ranged between about 81% and 96%. The precision of the method, expressed as relative standard deviation, was about 3% and the quantitation limit was around 9 ng g\(^{-1}\), which was far below the current specific migration limit (SML) set for BPA by the EU Commission (600 ng g\(^{-1}\)). The method was successfully applied to determine the BPA in the solid content of canned fruit salad, peaches in syrup, mango slices,
red peppers, sweetcorn, green beans, and peas. BPA was present at concentrations in the range from 7.8 to 24.4 ng g\(^{-1}\) in canned fruits and from 55 to 103 ng g\(^{-1}\) in canned vegetables.

High-speed countercurrent chromatography (HSCCC) has been applied extensively to the separation of synthetic dyes (Weisz and Ito, 2011). HSCCC is a liquid–liquid partition technique that does not involve the use of a solid support. One of the two immiscible liquid phases is retained in an Ito multilayer-coil column by centrifugal force while the other liquid phase is pumped through the rotating column.

The performance of three types of HSCCC instruments was assessed for their use by Weisz and Ito (2011) in separating components in hydrophilic and hydrophobic dye mixtures. The HSCCC instruments compared were

(i) A J-type coil planet centrifuge (CPC) system with a conventional multilayer-coil column, where the column holder revolves around the central axis of the centrifuge while rotating about its own axis at the same angular velocity and separates a broad range of hydrophobic and hydrophilic compounds, except for extremely polar compounds such as proteins and polysaccharides.

The system consisted of a column (three multilayer coils connected in series and made of 1.6 mm i.d. polytetrafluoroethylene (PTFE) tubing with a total capacity of ~320 mL) mounted on a rotating frame (centrifuge), a speed controller, and a Model 300 LC pump (Scientific Systems, State College, PA, USA). To this system they added a right-angle flow-switching valve to conveniently introduce into the column the stationary phase, a UV detector, a chart recorder, and a fraction collector.

(ii) A J-type CPC system with a spiral-tube assembly-coil column fitted with three spiral-tube assembly coils (CC Biotech LLC, Rockville, MD, USA) (Ito et al., 2008) for use as the column separating polar peptides, proteins, nucleic acids, and polysaccharides.

(iii) A cross-axis CPC system with a multilayer-coil column. It has an XL-type planetary motion where the column revolves around the vertical centrifuge axis while rotating about its horizontal axis at the same angular velocity.

The column consisted of four multilayer coils connected in series and made of 1.6 mm i.d. PTFE tubing with a total capacity of ~250 mL. The relevant parameters (Menet and Thiebaut, 1999) of the instrument are as follows: radius, \(r\), of the multilayer-coil holder, ~4.5 cm; distance between the two axes, \(R\), ~5 cm; and measure of the lateral shift of the multilayered-coil holder along its axis, \(L\), ~7.6 cm.

The hydrophilic dye mixture consisted of a sample of FD&C Blue No. 2 that contained mainly two isomeric components, 5,5′- and 5,7′-disulfonated indigo, in the ratio of ~7:1. The hydrophobic dye mixture consisted of a sample of D&C Red No. 17 (mainly Sudan III) and Sudan II in the ratio of ~4:1. The two-phase solvent systems used for these separations were 1-butanol/1.3 M HCl and hexane/acetonitrile. Each of the three instruments was used in two experiments for the hydrophilic dye mixture and two for the hydrophobic dye mixture, for a total of 12 experiments.
In one set of experiments, the lower phase was used as the mobile phase, and in the second set of experiments, the upper phase was used as the mobile phase. The results suggest that (a) the use of a J-type instrument with either a multilayer-coil column or a spiral-tube assembly column, applying the lower phase as the mobile phase, is preferable for separating the hydrophilic components of FD&C Blue No. 2, and (b) the use of a J-type instrument with multilayer-coil column, while applying either the upper phase or the lower phase as the mobile phase, is preferable for separating the hydrophobic dye mixture of D&C Red No. 17 and Sudan II.

3.5.1 Protein and Oil Recovery by Centrifugation from Various Plants

The processes involved in the separation and recovery of protein and oil include a demulsification step, followed by the separation of the aqueous and oil phases by centrifugation. Demulsification has been carried out in a number of different ways. The aim of demulsification is to promote or accelerate (in a thermodynamic sense) the mechanisms responsible for separation of the components.

The tendency of emulsified components to separate into distinctive phases can be understood from the second law of thermodynamics, as explained by Tadros (1989). The free energy for the formation of an emulsion is given by

\[ \Delta G_{\text{form}} = \gamma_{ow} \Delta a - \Theta \Delta S_{\text{Conf}} \]  

where \( \gamma_{ow} \) is the interfacial tension and \( \Delta a \) is the increase in interfacial area. During emulsification, the interfacial area (a) between the oil and water phases is greatly increased as a result of the oil droplet subdividing into much smaller units; this is accompanied by increased interfacial energy \( \gamma_{ow} \Delta a \).

Liquid–liquid separation, similar to emulsions, occurs in the dairy industry where milk is fed into the bowl of a vertical continuous liquid centrifuge and separated into skimmed milk and cream.

Virgin olive oil is extracted in olive oil mills with pressure, centrifugation, and percolation systems by using different apparatus driven by physical forces, which, when correctly exerted on olive paste, enables the separation of the different phases of olives: liquid and solid. In the past, the percolation system was coupled with pressures; at present it is coupled with the centrifugal decanter (Mascolo 1978; Martinez-Suarez et al., 1974).

Centrifugation is a worldwide system based on the centrifugal force applied on olive paste, diluted with lukewarm water. The dilution increases the difference between the specific weights of the immiscible liquids (oil and vegetable water) and the solid matter.

Di Giovacchino et al. (2002) reported that the separation of oil from the solid and liquid phases of olive paste is performed by using pressure, percolation, or centrifugation. All systems may provide good-quality oil if olive fruits are sound and at the correct ripeness, but the centrifugation system helps to avoid or reduce the risk of an organoleptic contamination. The new centrifugal decanters operating without adding
water (or only a minimal amount of water) to olive paste save heat energy, and the oils obtained are more fruity and have a higher content of natural phenolic antioxidants.

3.5.2 CENTRIFUGE EQUIPMENT

The simplest form of centrifuge consists of a bowl spinning about a vertical axis. Liquids, or liquids and solids, are introduced into this, and under centrifugal force, the heavier liquid or particles pass to the outermost regions of the bowl, while the lighter components move toward the center.

The main categories are liquid–liquid centrifuges, centrifugal clarifiers for removal of small amounts of solids, and desludging or dewatering centrifuges.

3.5.2.1 Liquid–Liquid Centrifuges

The simplest type of equipment is the tubular bowl centrifuge. It consists of a vertical cylinder (or bowl), typically 0.1 m in diameter and 0.75 m long, which rotates inside a stationary casing at between 15,000 and 50,000 rev min$^{-1}$ depending on the diameter.

If the feed is all liquid, then collection pipes can be fitted accordingly to separate the heavier and the lighter components. Various arrangements are used for this. Centrifuge action is analogous to gravity settling, with the various weirs and overflows acting in the same way as in a settling tank even though the centrifugal forces are much greater than gravity. In liquid/liquid separation centrifuges, conical plates are arranged, giving smoother flow and better separation.

Better separation is obtained by thinner layers of liquid formed in the disk bowl centrifuge. Here, a cylindrical bowl, 0.2–1.2 m in diameter, contains a stack of inverted metal cones that have a fixed clearance of 0.5–1.27 mm and rotate at 2000–7000 rev min$^{-1}$.

Whereas liquid phases can easily be removed from a centrifuge, solids present much more of a problem.

3.5.2.2 Centrifugal Clarifiers

The simplest solid–liquid centrifuge is a solid bowl clarifier, which is a rotating cylindrical bowl, 0.6–1.0 m in diameter. Liquor, with a maximum of 3% w/w solids, is fed into the bowl and the solids form a cake on the bowl wall (Fellows, 2000).

The mixture to be clarified enters the rotor through a centrally arranged feed tube. The distributor accelerates the product and conveys it into the separating space to the disk stack where the actual separation of solids and liquid takes place. In a centrifugal separator, the separated phases are discharged under pressure by centripetal pump.

3.5.2.3 Disk Separators with Solid-Wall Bowl

By fitting a large number of conical disks separated by spacers between 0.3 and 2 mm, a substantially larger equivalent clarification area is achieved with the same bowl volume compared to chamber bowl separators. The equivalent clarification area is the product of the sum of the geometric surfaces of the disks multiplied with the acceleration due to gravity.
Solid-wall separators are available in two versions:

- Separators of two liquids dissolved in one another
- Clarifiers for the separation of solids from liquids

Solid-wall separators are used almost exclusively for the separation of liquid mixtures with little or no solids content since the separated solids can only be removed manually. Example of a disk wall separator is given in Figure 3.9.

### 3.5.2.4 Chamber Bowl Separators

Chamber bowl separators are solid bowl centrifuges with annular inserts (chambers). They are used for clarification, that is, the separation of solids from suspensions. The suspension flows through the individual chambers from the inside to the outside, whereby the solids deposit in the chambers. The clarified liquid discharges under gravity over an overflow weir (regulating ring) or is discharged under pressure by means of a centripetal pump.

The machines must be shut down to empty chamber bowl separators; the separated solids are removed manually.
Application criteria for chamber bowl separators are

- They are ideal for polishing suspensions, that is, for separating smallest solid contents since they feature excellent flow characteristics.
- Owing to the long retention time in the centrifugal field, the separated solids are recovered in a very compact state.
- Chamber bowl separators cannot be used when processing with centrifuges equipped with an automatic discharge system due to insufficient shear stability or excessive erosive effect.

3.5.2.5 Self-Cleaning Disk Separators

The disk separators with self-cleaning bowls are equipped with a stack of conical disks to create a large equivalent clarification area within a relatively small bowl volume. Separators with self-cleaning bowls are able to periodically discharge the separated solids at full speed. For this purpose, several ports are spaced evenly around the bowl periphery. These ports are opened and closed by means of a movable sliding piston located in the bowl bottom. The opening mechanism is actuated hydraulically. Water is normally used as a control medium; in special cases, low-viscous organic fluids can also be used.

This opening mechanism enables both partial ejections and total ejections. Total ejections involve discharging the entire contents of the bowl with closed feed valve. In the case of a partial ejection, by contrast, only part of the bowl content is ejected with an open feed valve.

3.5.2.6 Westfalia Separator® Hydrostop

Westfalia Separator hydrostop is a special system developed by GEA Westfalia Separator (2013a), which can be adjusted to specific requirements in terms of solid concentration exactly and reproducibly. This patented ejection system makes it possible to optimize the ejection cycle to the shortest possible time.

The Westfalia Separator hydrostop system reduces the actual ejection time to less than a tenth of a second and permits partial ejections to be performed in a 30 s rhythm. This assures that even small volumes of 1.5 to 2 L are discharged reproducibly with a margin of error of less than 10%. This innovative technology enables precise, fast ejections and, hence, significantly higher and qualitatively better yields (http://www.westfalia-separator.com/products/separators/self-cleaning-disk-separators.html).

Feeds that contain a higher solid content are separated using nozzle centrifuges or valve discharge centrifuges. These are similar to disk bowl types, but the bowls have a biconical shape.

In the nozzle type, solids are continuously discharged through small holes at the periphery of the bowl and are collected in a containing vessel. In the valve type, the holes are fitted with valves that periodically open for a fraction of a second to discharge the accumulated solids.

Nozzle separators are continuously operating disk centrifuges. They are built as centrifugal clarifiers and separators and are more solids orientated than self-cleaning separators. When they are configured to function as clarifiers, they are called concentrators. They are used to thicken solids from suspensions.
Nozzle separators are absolute specialists in the processing of fermentation products in biotechnology and the pharmaceutical and food industries. The separated solids are discharged continuously through nozzles fitted at the bowl periphery. The nozzle separators are equipped with a hydraulically operated ejection system, which enables both partial and total ejections to be triggered during product separation.

The advantage: When processing difficult products, the separating time to the next CIP cycle can be extended. The time-controlled or process-dependent ejections additionally optimize cleaning of nozzles, bowl, and disks.

In addition to models equipped with standard nozzles, GEA Westfalia Separator also offers separators featuring patented special Westfalia Separator Viscon® nozzles (viscosity-controlled nozzles). In contrast to standard nozzles, they guarantee a virtually constant biomass concentration by means of an automatic, internal concentration regulation, even in the case of fluctuating feed volumes and concentrations. Volume streams of over 300 m³ h⁻¹ can be processed with the largest models in this series.

Centrifugal clarifiers are used to treat oils, juices, beer, and starches and to recover yeast cells. They have capacities up to 300,000 L h⁻¹. Centrifugal clarifiers can also be used in the manufacturing of whey and whey cream.

Whey is defined as the liquid that forms in cheese-making after the casein and fat have been separated when the milk clots. Continued processing of whey can lead to the production of whey protein concentrate, whey concentrate, whey powder, and lactose.

Separators can also be used to recover valuable constituents such as cheese dust or lactalbumin. Lactalbumin is a mixture of various whey proteins. It is traditionally recovered by heat denaturation of wheys from various origins, primarily from cheese wheys. Once the lactalbumin has become insoluble, it can be centrifuged by separators and dried in a spray-drying tower to form a powder. Lactalbumin has a high nutritional value.

The obtaining and further processing of unwanted products from whey can also be demonstrated by the example of cheese dust. Cheese dust is defined as very fine cheese particles of a few μm to 1 mm in size which are formed during the cutting, agitating, and pumping of curd. These protein particles get into the whey during the separation of curd and whey or when draining the cheese molds and pressing them. Many large dairies separate the cheese dust from the whey with clarifiers from GEA Westfalia Separator and then use decanters to remove more moisture from the concentrate thus obtained to form a free-flowing cheese mass with a dry matter of around 40%. This mass can then be made into processed cheese.

Westfalia separators can also be used in warm or cold milk skimming. Mechanical skimming by centrifugal force is necessary to separate the raw milk into cream and skimmed milk. After separation, both are mixed together again in a certain ratio (depending on whether low-fat milk, full-fat milk, or cream is to be produced) until the desired fat content is set.

Most dairies work with warm milk skimming; the raw milk is first heated and then skimmed warm. Because of the higher temperature, there is a significant difference in density between cream and skimmed milk and this has a positive impact on the skimming precision of a separator.
However, cold milk skimming is on the rise, especially in the United States, Mexico, Australia, and New Zealand. Here, the operating temperature is between 4°C and 20°C. This means lower energy consumption and thus reduced production costs for the dairy when compared with the previous skimming temperatures of 52–55°C.

GEA Westfalia’s procool separator, the first cold milk centrifuge to use a belt drive and therefore consume less energy, supports cold milk skimming.

In liquid/solid separation, stationary ploughs cannot be used as these create too much disturbance of the flow pattern on which the centrifuge depends for its separation. One method of handling solids is to provide nozzles on the circumference of the centrifuge bowl as illustrated in Figure 3.10. These nozzles may be opened at intervals to discharge accumulated solids together with some of the heavy liquid. Alternatively, the nozzles may be open continuously relying on their size and position to discharge the solids with as little as possible of the heavier liquid. These machines thus separate the feed into three streams—light liquid, heavy liquid, and solids—the solids carrying with them some of the heavy liquid as well. Similar to the operation of disk nozzles, there are disks with intermittent discharge.

Another method of handling solids from continuous feed is to employ telescoping action in the bowl: sections of the bowl moving over one another and conveying the solids that have accumulated toward the outlet (Earle, 1983).

The horizontal bowl with scroll discharge, centrifuge, as illustrated in Figure 3.11, can discharge continuously. In this machine, the horizontal collection scroll (or screw) rotates inside the conical-ended bowl of the machine and conveys the solids with it, while the liquid discharges over an overflow toward the center of the machine and at the opposite end to the solid discharge. The speed of the scroll, relative to the bowl, must not be great. For example, if the bowl speed is 2000 rev min\(^{-1}\), a suitable speed for the scroll might be 25 rev min\(^{-1}\) relative to the bowl, which would mean a scroll speed of 2025 or 1975 rev min\(^{-1}\) (Coulson et al., 1978; Foust et al., 1980; Heldman and Hartel, 1997a,b; McCabe et al., 1985; Trowbridge 1962).

Specific applications of centrifuges are described by Hemfort (1984) for the fermentation industries and by Hemfort (1983) for the food industry.

Finally, tubular bowls exist as shown in Figure 3.12.
3.5.2.7 Desludging, Decanting, or Dewatering Centrifuges

Feeds with high solid contents are separated using desludging centrifuges, including conveyor bowls, screen conveyors, baskets, and reciprocating conveyor centrifuges. In the conveyor bowl centrifuge, the solid bowl rotates up to 25 rev min$^{-1}$ faster than the screw conveyor. This causes the solids to be conveyed to one end of the centrifuge, whereas the liquid fraction moves to the other larger-diameter end (Fellows, 2000).

The solids are relatively dry compared with other types of equipment. The screen conveyor centrifuge has a similar design but the bowl is perforated to remove the liquid fraction. The reciprocating conveyor centrifuge is used to separate fragile solids (e.g., crystals from liquor).

3.6 FILTRATION

Filtration is the phenomenon when a fluid is subject to a force that moves it past the retained particles. The particles suspended in the fluid, which will not pass through the apertures, are retained and build up to form a cake of increasing thickness, the so-called filter cake. The fine apertures necessary for filtration are provided by fabric filter cloths, meshes and screens of plastics or metals, or by beds of solid particles.

Rate of filtration = driving force/resistance
Resistance arises from the filter cloth, mesh, or bed, and to this is added the resistance of the filter cake as it accumulates. The filter cake resistance is obtained by multiplying the specific resistance of the filter cake, that is, the resistance per unit thickness, by the thickness of the cake. The resistances of the filter material and precoat are combined into a single resistance called the filter resistance.

Filtration can be classified by the following:

1. Driving force
   The filtrate is induced to flow through the filter medium by hydrostatic head (gravity), pressure applied upstream of the filter medium, vacuum or reduced pressure applied downstream of the filter medium, or centrifugal force across the medium.

2. Filtration mechanism
   When solids are trapped within the pores or body of the medium, it is termed depth, filter-medium, or clarifying filtration.

3. Objective
   The process goal of filtration may be dry solids, clarified liquid (filtrate), or both. Good solid recovery is best obtained by cake filtration, while clarification of the liquid is accomplished by either depth or cake filtration.

4. Operating cycle
   Filtration may be intermittent (batch) or continuous. Batch filters may be operated with constant-pressure driving force, at constant rate, or in cycles that are variable with respect to both pressure and rate. Batch cycle can vary greatly, depending on filter area and solid loading.

5. Nature of the solids
   Cake filtration may involve an accumulation of solids that is compressible or substantially incompressible, corresponding roughly in filter-medium filtration to particles that are deformable and to those that are rigid. The particle or particle-aggregate size may be of the same order of magnitude as the minimum pore size of most filter media (1–10 μm and greater), or may be smaller (1 μm down to the dimension of bacteria and even large molecules) (Perry, 1999).

It is convenient to express the filter resistance in terms of a fictitious thickness of filter cake. This thickness is multiplied by the specific resistance of the filter cake to give the filter resistance. Thus, the overall equation giving the volumetric rate of flow \( \frac{dV}{dt} \) is

\[
\frac{dV}{dt} = \frac{(A \cdot \Delta P)}{R}
\]  

(3.6)

As the total resistance is proportional to the viscosity of the fluid, we can write

\[
R = 1 + \Theta (L_c + L)
\]  

(3.7)
where $R$ is the resistance to flow through the filter, $\mu$ is the viscosity of the fluid, $r$ is the specific resistance of the filter cake, $L_c$ is the thickness of the filter cake, and $L$ is the fictitious equivalent thickness of the filter cloth and precoat, $A$ is the filter area, and $\Delta P$ is the pressure drop across the filter.

If the rate of flow of the liquid and its solid content are known and assuming that all solids are retained on the filter, the thickness of the filter cake can be expressed by

$$L_c = \frac{wV}{A}$$

where $w$ is the fractional solid content per unit volume of liquid, $V$ is the volume of fluid that has passed through the filter, and $A$ is the area of filter surface on which the cake forms.

The resistance can then be written as

$$R = \mu r\left[\frac{wV}{A} + L\right]$$

and the equation for flow through the filter, under the driving force of the pressure drop is then (Earle, 1983)

$$\frac{dV}{dt} = \frac{A\Delta P}{\mu r\left[\frac{wV}{A} + L\right]}$$

Moreover, Perry (1999) reported that in cake or surface filtration, there are two areas: continuous filtration, in which the resistance of the filter cake (deposited process solids) is very large with respect to that of the filter media and filtrate drainage, and batch pressure filtration, in which the resistance of the filter cake is not very large. Batch pressure filters are generally fitted with heavy, tight filter cloths plus a layer of precoat, and continuous filters, except for precoats, use relatively open cloths that offer little resistance compared to that of the filter cake.

Simplified theory for both batch and continuous filtration is based on Hagen–Poiseuille equation:

$$\frac{dV}{dt} = \frac{A\Delta P}{\mu \left(awV/A + r\right)}$$

where $V$ is the volume of filtrate collected, $t$ is the filtration time, $A$ is the filter area, $P$ is the total pressure across the system, $w$ is the weight of cake solids/unit volume of filtrate, $\mu$ is the filtrate viscosity, $a$ is the cake-specific resistance, and $r$ is the resistance of the filter cloth plus the drainage system.

In the filtration process, the components that are rejected accumulate near the membrane surface. This phenomenon is defined as concentration polarization and has significant consequences, especially in UF. The other phenomenon that can occur is the formation of the so-called gel layer, which acts as an extra hydraulic resistance. This also results in a lowering of the flux.

Fouling can become more important if the components in the gel layer react with each other and form a dense cross-linked layer on the top of the membrane (de Bruijn et al., 2003).
3.6.1 Constant-Rate Filtration

In the early stages of a filtration cycle, it frequently happens that the filter resistance is large relative to the resistance of the filter cake because the cake is thin. Under these circumstances, the resistance offered to the flow is virtually constant and so filtration proceeds at a more or less constant rate.

3.6.2 Constant-Pressure Filtration

Once the initial cake has been built up, and this is true of the greater part of many practical filtration operations, flow occurs under a constant-pressure differential. Under these conditions, the term \( \Delta P \) in Equation 3.10 is constant and so

\[
\mu_r [w(V/A) + L] dV = A \Delta P dt
\]

and integrating from \( V = 0 \) at \( t = 0 \), to \( V = V \) at \( t = t \)

\[ \mu_r [w(V^2/2A) + LV] = A \Delta P t \]

rewriting this

\[
t (V/A) = (\mu_r w/2\Delta P) \times (V/A) + \mu_r L / \Delta P
\]

Equation 3.14 is useful because it covers a situation that is frequently found in a practical filtration plant. It can be used to predict the performance of filtration plant on the basis of experimental results. If a test is carried out using constant pressure, collecting and measuring the filtrate at measured time intervals, a filtration graph can be plotted of \( t/(V/A) \) against \( (V/A) \) and from the statement of Equation 3.14, it can be seen that this graph should be a straight line. The slope of this line will correspond to \( \mu_r w/2\Delta P \) and the intercept on the \( t/(V/A) \) axis will give the value of \( \mu_r L / \Delta P \). Since, in general, \( \mu \), \( w \), \( \Delta P \), and \( A \) are known or can be measured, the values of the slope and intercept on this graph enable \( L \) and \( r \) to be calculated (Earle, 1983; Perry, 1999).

In constant filtration, we assume that the resistance of the filter cloth plus filtrate drainage is negligible compared to the resistance of the filter cake and also that both pressure drop and specific cake resistance remain constant throughout the filter cycle.

\[
W = \frac{2wPt}{\mu \left( awV/A + r \right)}
\]

and

\[
V_f = \frac{2Pt}{\mu aw}
\]

where \( W \) is the weight of dry filter cake solids/unit area, \( V_f \) is the volume of cake formation filtrate/unit area, \( V_w \) is the volume of cake wash filtrate/unit area, \( t \) is the
cake formation time, and $N$ is the wash ratio, the volume of cake wash/volume of liquid in the discharged cake.

### 3.6.3 FILTER CAKE COMPRESSIBILITY

With some filter cakes, the specific resistance varies with the pressure drop across it. This is because the cake becomes denser under higher pressure and so provides fewer and smaller passages for flow. The effect is known as the compressibility of the cake. Soft and flocculent materials provide highly compressible filter cakes, whereas hard granular materials, such as sugar and salt crystals, are little affected by pressure. To allow for cake compressibility, the following empirical relationship has been proposed:

$$r = r' \Delta P^s$$  \hspace{1cm} (3.17)

where $r$ is the average specific cake resistance under pressure $P$, $\Delta P$ is the pressure drop across the filter, $r'$ is the specific resistance of the cake under a pressure drop of 1 atm and is a constant determined largely by the size of the particles forming the cake, and $s$ is a constant for the material, called cake compressibility, varying from 0 for rigid, incompressible cakes, such as fine sand and diatomite, to 1.0 for very highly compressible cakes. For most industrial slurries, $s$ lies between 0.1 and 0.8.

This expression for $r$ can be inserted into the filtration equations, such as Equation 3.14, and values for $r'$ and $s$ can be determined by carrying out experimental runs under various pressures (Perry, 1999; Earle, 1983).

### 3.6.4 FILTER MEDIA

The selection of filter media depends on the optimization of the following factors (Perry, 1999):

1. Ability to bridge solids across its pores quickly after the feed is started (i.e., minimum propensity to bleed)
2. Low rate of entrapment of solids within its interstices (i.e., minimum propensity to blind)
3. Minimum resistance to filtrate flow (i.e., high production rate)
4. Resistance to chemical attack
5. Sufficient strength to support the filtering pressure
6. Acceptable resistance to mechanical wear
7. Ability to discharge cake easily and cleanly
8. Ability to conform mechanically to the filter with which it will be used
9. Minimum cost

### 3.6.5 ENHANCED FILTRATION

Enhanced filtration is used by the Coca Cola company to treat 100% of raw water by using one of the following processes:

- Coagulation/flocculation followed by granular media filtration (conventional chemical treatment) Fe or Al coagulant
Coagulation/flocculation is the combination of two processes working together to destabilize stable particulate suspensions in water and combine these destabilized particles into particles large enough for sedimentation and filtration. Another name for stable particulate suspensions is “colloidal suspension.” Coagulation is the process of particle destabilization. Flocculation is the process of combining these destabilized particles. A suspension is a system in which very small particles (solid, semisolid, or liquid) are more or less uniformly dispersed in a liquid or gaseous medium. If the particles are small enough to pass through a filter membrane, between 1 nm and 1 μm, the system is called a “colloidal suspension.”

Contributing to the stability of the suspension is that, in general, in natural waters the particles in the suspension carry a negative surface charge. These negative surface charges interact to push (repulse) the particles away from each other (Coca Cola Company, 2003).

Particulate suspensions commonly removed by coagulation/flocculation include:

- Clay, silt-based turbidity, natural organic matter (including disinfection by-product precursors), microbial matter (including bacteria, yeast, molds, virus, protozoa), metals, synthetic organic chemicals, iron, manganese, off-taste causing compounds, odor-causing compounds.

There are two major groups of flocculants:

- Inorganic flocculants
- Organic flocculants (commonly referred to as coagulant aids)

There are two main types of inorganic flocculants: cationic iron flocculants and cationic aluminum flocculants.

The two common forms of cationic iron flocculants are ferrous (Fe^{2+}), such as ferrous sulfate, and ferric (Fe^{3+}), such as ferric sulfate or ferric chloride.

The two common forms of cationic aluminum flocculants are aluminum sulfate and polyaluminum chloride, both Al^{3+}.

There are three steps in coagulation/flocculation:

1. Coagulant formation
   The flocculant is added to the water that is to be treated and immediately dissolves and hydrolyzes.

2. Coagulation (also called charge neutralization or particle destabilization)
   The coagulant cation’s positive charge neutralizes the negative surface charges typically carried by the suspended particulates. This neutralization step takes place in fractions of a second and allows the suspended particles to come close enough together to begin building a floc.

3. Flocculation (also called particle aggression) (Figure 3.13)
   Particle charge is now no longer important. A gelatinous flocc begins to build and becomes a “sticky” trap for material suspended in the water.

Iron-based flocculants:

- Ferrous sulfate \{FeSO_4 \times 7H_2O\}
- Ferrous (Fe^{2+}) must be oxidized to the (Fe^{3+}) valence state
Centrifugation–Filtration

Ferric chloride \( \text{FeCl}_3 \)
Ferric sulfate \( \text{Fe}_2(\text{SO}_4)_3 \)
Ferric hydroxide \( \text{Fe}_2(\text{OH})_3 \) coagulant

Aluminum-based flocculants:

Aluminum sulfate \( \text{Al}_2(\text{SO}_4)_3 \times 14\text{H}_2\text{O} \)
Polyaluminum chloride (a partially hydrolyzed salt)
Aluminum hydroxide \( \text{Al}_2(\text{OH})_3 \) coagulant

Ferric iron coagulants are recommended since they are insoluble over a much wider range of pH, and as a result, offer significant operational advantages.

Coagulant aids are very sensitive to tiny changes in the water’s quality, so they can unbalance the treatment process. If coagulant aids remain in the water after treatment, they also can affect the final product. Many countries have legal limits on doses of coagulant aids because they may be toxic.

Granular media filtration is the last step in enhanced filtration using coagulation/filtration.

Water filtration separates suspended matter from water by passing it through porous media. Coagulation destabilizes particles and flocculation makes them cluster into “macrofloc” particles. These flocs are large enough to be retained by the porous media as the water flows through it.

There are four types of media for enhanced filtration: anthracite, silica sand, garnet, and gravel.

Manufacturers build granular media filters to operate in two main conditions: downflow or upflow under pressure and under gravity feed. Filters that operate under pressure in upflow or downflow usually are of lined steel or stainless steel, so they can withstand the pressure and have typical cylindrical shape. Filters operating under
Gravity feed usually are of lined steel and often are rectangular. They can use single, dual, or multiple media depending on the source water’s characteristics (Figure 3.14).

Other filtration processes include direct filtration (injection of flocculant in-line followed by static mixing and deep bed multimedia filtration), which is only used if raw water meets the following recommended quality criteria:

- Turbidity: <5 NTU
- KMnO₄: <5 ppm
- Iron: <0.3 ppm
- Manganese: <0.05 ppm
- TOC: <3 ppm (total organic carbon)

UF (<20,000 MWCO) can also be used with ion exchange.

NF is another filtration method and RO using CA membrane or PA (Figure 3.15).

**Alkalinity reduction** If necessary (if alkalinity > 85 mg L⁻¹), a system can be designed to reduce enough alkalinity in raw water so that it meets the specifications for treated water.

One of the following processes can be employed to reduce alkalinity: hydrated lime treatment with lime Ca(OH)₂, ion exchange, NF, RO, or ED.

**Disinfection (for control of microorganisms)** Sodium hypochlorite disinfects to control microorganisms remaining in source water after it passes through enhanced filtration. Sodium hypochlorite (NaOCl) comes as a liquid, at a pH of about 11 for stability.
When sodium hypochlorite dissolves in water, calcium carbonate and other insolubles precipitate. Coagulation, flocculation, and filtration then remove the insolubles from the water.

As sodium hypochlorite reacts with water, it forms hypochlorous acid (HOCl)—the best source of free chlorine for disinfection.

Chlorine disinfection controls microorganisms remaining in the source water after the enhanced filtration stage.

Hypochlorous acid (HOCl) and hypochlorite ion (OCl\(^{-}\)) both are known as free chlorine, but HOCl more effectively kills microorganisms.

Chlorine can also be used to disinfect 100% of raw water assuring the water contacts the chlorine for at least 30 min. Ultraviolet light (UV) can only be used for supplemental disinfection after enhanced filtration systems that use membranes. The UV lamp should produce at least 40,000 \(\mu\)W cm\(^{-2}\) on the 254 nm wavelength at the end of life. Residual chlorine of at least 1 ppm provides the primary disinfection.

**Carbon purifiers (to control taste-odor)** Carbon purifiers can be employed to treat 100% of raw water by using the following specifications: bed depth of at least 1.5 m, 50% freeboard, stainless-steel type 316, and a flow rate of 140 L min\(^{-1}\) m\(^{-3}\).

Through adsorption, granular activated carbon reduces or removes certain chemicals and materials remaining after other treatment.

Organic materials that are regulated are materials that cause off-tastes and odors, chlorine and ozone.
Bituminous coal works best for water treatment. It filters various materials in water because it is very hard, contains little fines or dust, and has a good pore size distribution.

Polishing filtration (to control appearance) It is used by removing small particles of carbon in treated water.

Treated water passes through filters with pore size of 5–20 microns absolute. (This pore size results in at least 3-log removal of 30 μm particles or larger with a beta factor of 1000.) Filtering media compatible with steam or chlorine sanitation are used. Filter elements are usually single-ended and sealed by double O-rings (Coca Cola, 2003).

Total trihalomethane (TTHM) needs to be covered by strict regulations. TTHM is the sum of chloroform (CHCl₃), bromoform (CHBr₃), dichlorobromomethane (CHCl₂Br), and dibromochloromethane (CHClBr₂). In Europe, the following countries have a tighter spec on TTHM: Germany: 10 ppb, Austria: 30 ppb, Italy: 30 ppb, Sweden: 50 ppb.

3.6.6 Filtration Equipment

The basic requirements for filtration equipment are: mechanical support for the filter medium, flow accesses to and from the filter medium, and provision for removing excess filter cake.

In some instances, washing of the filter cake to remove traces of the solution may be necessary. Pressure can be provided on the upstream side of the filter, or a vacuum can be drawn downstream, or both can be used to drive the wash fluid through.

3.6.6.1 String Discharge Filter

A system of endless strings or wires spaced about 13 mm apart pass around the filter drum but are separated tangentially from the drum at the point of cake discharge, lifting the cake off as they leave contact with the drum. The strings return to the drum surface guided by two rollers, the cake separating from the strings as they pass over the rollers (Figure 3.16).

3.6.6.2 Plate-and-Frame Filter Press: Recessed Chamber Filter Press and/or Diaphragm (Membrane) Filter Press

In the plate-and-frame filter press, a cloth or mesh is spread out over plates that support the cloth along ridges, but at the same time leave a free large area below the cloth for flow of the filtrate. The plates with their filter cloths may be horizontal or vertical with a number of plates operated in parallel to give sufficient area.

Perry (1999) defined this press as an alternate assembly of plates covered on both sides with a filter medium, usually a cloth, and hollow frames that provide space for cake accumulation during filtration. The frames have feed and wash manifold ports, while the plates have filtrate drainage ports.

Filter cake builds up on the upstream side of the cloth that is the side away from the plate. In the early stages of the filtration cycle, the pressure drop across the cloth is small and filtration proceeds at a more or less constant rate. As the cake increases, the process becomes more and more a constant pressure, and this is the
case throughout most of the cycle. When the available space between successive frames is filled with cake, the press has to be dismantled and the cake scraped off and cleaned, after which a further cycle can be initiated.

Plates, which may be rectangular or circular, are supported on a central hollow shaft for the filtrate and the whole assembly enclosed in a pressure tank containing the slurry (Figure 3.17). Filtration can be done under pressure or vacuum. The advantage of vacuum filtration is that the pressure drop can be maintained while the cake is still under atmospheric pressure and so can be removed easily. The disadvantages are the greater costs of maintaining a given pressure drop by applying a vacuum and the limitation on the vacuum to about 80 kPa maximum. In pressure filtration, the pressure driving force is limited only by the cost and the mechanical strength of the equipment.

The above plate-and-frame filter press can be used to treat wastewater with high solids. These presses provide excellent solids retention, producing 35–50% solids in the filter cake. These presses routinely outperform rotary vacuum, horizontal vacuum, and belt filter presses.

Plate-and-frame filter presses are dewatering machines that utilize pressure (60–80 psi, typically) to remove the liquid from a liquid–solid slurry. They are particularly suited for low solids (<2% solids), or solids composed of fines (~200 mesh); however, they will essentially dewater many combinations of particle size distribution and percent solid slurries.

The basic operation of a plate-and-frame filter press in Figure 3.17 shows the feed entering the press at the bottom of the plate, using a pump suitable for pumping up to 80–90 psi. Then, the feed travels the path of least resistance (up between the filter plates), which has filter media inserted between the plates, and the void between the plates is filled with the slurry, as the liquid passes through...
the filter media, and travels up to the outlet port at the top of the plate. This liquid is referred to as the “filtrate,” and is discharged from the press. The solids remain in the void between the plates, until the plates discharge the filtered solids (http://www.beckart.com/wastewater_treatment/filter_presses.php).

Large plate-and-frame filter presses have mechanical “plate shifters,” to move the plates, allowing the rapid discharge of the solids stuck in between them. Also, they have the capability of blowing compressed air into the plate, to dry the cake, and to aid in its discharge. Typical capacities for a plate-and-frame filter will depend upon the solids being dewatered.

The filter press may also be used as a “polishing” filter to remove minute quantities of solids from an influent stream. In these applications, the press is not sized for the quantity of solid-holding capacity but for maximum filtration area and hydraulic throughput. When used as a polishing filter, generally a dry filter cake is not developed. Rather, when throughput flow rates drop to an unacceptable level, the cycle is ended.

The last most common usage of the filter press is as a “variable volume” filter through the use of a filter plate known as a diaphragm or membrane plate. This type of plate has a flexible drain field, which when sealed around the edges forms an integral bladder or diaphragm that may be inflated to physically press additional liquid from the filter cake. This process can significantly reduce the typical elapsed time for a press cycle and produce a dryer cake product, or more uniform cake dryness from cycle to cycle (http://www.water.siemens.com/en/products/sludge_biosolids_processing/filter_press/Pages/dewatering_systems_generic_what_is_a_filter_press.aspx).
The filter press is made up of two principal components: the skeleton and the filter pack.

The skeleton holds the filter pack together against the pressures developed internally during the filtration process and includes a stationary and follower head, hydraulics, and manifold.

The filter pack is where the actual liquid/solid separation process takes place. The pack consists of a series of filter elements that form a series of chambers when held together in the press skeleton. Each chamber wall has a series of raised cylinders or “pips,” which are then covered with a porous cloth medium. These pips form a flow path for the liquid draining from the press.

The prime function of the filter media is to provide a porous support structure for the filter cake as it develops and builds. Initially, some solids may pass through the cloth media, causing a slight turbidity in the filtrate, but gradually the larger particles within the slurry begin to bridge the openings in the media, reducing the effective opening size. This allows smaller particles to bridge these reduced openings initiating the cake filtration process. Once a layer of solid particles achieves 1–2 mm in thickness, this “precoat” layer serves to separate out finer and finer particles as the cake builds in thickness, yielding a filtrate that is very low in turbidity.

The pressure behind the slurry (typically 100 psi, but up to 900 psi [7–60 bar]) is provided by a feed pump—sometimes a positive displacement or centrifugal pump. It is the existence of this pressure differential (between the feed pressure and the gravity discharge), not just the feed pump pressure, that causes the filtering action to occur. Solids within the slurry will flow to the area of cake development with the lowest pressure differential, resulting in a filter cake that builds uniformly over the drain field on either side of the chamber walls.

Two wash techniques are used in plate-and-frame filter presses. In simple washing, the wash liquor follows the same path as the filtrate. If the cake is not extremely uniform and highly permeable, this type of washing is ineffective in a well-filled press. A better technique is thorough washing, in which the wash is introduced to the faces of alternate plates (with their discharge channels valved off). The wash passes through the entire cake and exits through the faces of the other plates (Perry, 1999).

3.6.6.2.1 Rotary Filters

In rotary filters, the flow passes through a rotating cylindrical cloth from which the filter cake can be continuously scraped. Either pressure or vacuum can provide the driving force, but a particularly useful form is the rotary vacuum filter. In this, the cloth is supported on the periphery of a horizontal cylindrical drum that dips into a bath of the slurry. Vacuum is drawn in those segments of the drum surface on which the cake is building up. A suitable bearing applies the vacuum at the stage where the actual filtration commences and breaks the vacuum at the stage where the cake is being scraped off after filtration. Filtrate is removed through trunnion bearings. Rotary vacuum filters are expensive, but they do provide a considerable degree of mechanization and convenience. A rotary vacuum filter is illustrated diagrammatically in Figure 3.18.

Vacuum filters can be divided into bottom feed (precoat and drum filters), side feed (disk filters), and top feed (belt, tray, table, and tilting pan filters).
There are two concepts in the layout design of vacuum filters: barometric leg layouts and floor-mounted layouts. In barometric leg layouts, the filter is mounted 8–9 m above ground level to ensure that the liquid level in the leg will not flood the filtrate receiver. This applies to liquids with a specific gravity of 1; however, for heavier liquids, the filter elevation with regard to the ground level may be lowered. Barometric leg layouts require, therefore, a structure for the filter, but the advantage is that the filtrate pump operates under a positive suction head. On the other hand, floor-mounted layouts require a receiver-mounted filtrate pump designed to effectively deliver the filtrate under full vacuum on the suction side.

The DORR-OLIVER Horizontal Pan Filter is a continuous vacuum filter designed for economic filtration of granular, fibrous and coarse fast-settling solids, including cake washing and drying, if required. The DORR-OLIVER Horizontal Pan Filter provides a circular filtration surface (“pan”) rotating in a horizontal plane. Multiple filter designs, filter sizes, and process components are available, suitable for a wide range of industrial applications and process needs.

The unique filter design allows for cake washing in countercurrent mode thereby achieving highest cake washing efficiency at reduced wash media consumption. The cake moisture is effectively reduced by means of the applied vacuum and additional methods if required before it is removed by a wear-resistant cake discharge scroll. The filtration surface can be dressed with a wide range of textile and metal type of filter media, thereby using most advanced cloth fixing methods, including cassette designs. (http://www.flsmidth.com/en-US/Products/Product + Index/All + Products/Vacuum + Filtration/Horizontal + Pan + Filter/DORR-OLIVER + Horizontal + Pan + Filter).

All drum filters (except the single-compartment filter) utilize a rotary-valve arrangement in the drum-axis support trunnion to facilitate removal of filtrate and wash liquid and to allow introduction of air or gas for cake blowback if needed. The valve controls the relative duration of each cycle as well as providing “dead” portions of the cycle through the use of bridge blocks (Perry, 1999).
### 3.6.6.3 Centrifugal Filters

Centrifugal force is used to provide the driving force in some filters. These machines are centrifuges fitted with a perforated bowl that may also have filter cloth on it. Liquid is fed into the interior of the bowl and under the centrifugal forces, it passes out through the filter material.

During filtration, the filter vessel is fed under pressure; the filtrate passes through the plates and out through the shaft. The filter cake forms on the upper side of the filter elements. After filtration, the remaining feed in the vessel is either drained or filtered via the scavenge system. The cake may then be washed or dried by an appropriate heated gas. Spinning the entire stack at moderate speeds generates a centrifugal force that discharges the cake. The cake can be discharged in slurry or dry form. This is illustrated in Figure 3.19.

#### FIGURE 3.19 SeitzSchenk Centrifugal Discharge Filter. (Courtesy of Pall. With permission.)

1. Filter vessel with sight glass
2. Hollow filter shaft
3. Filter plate
4. Scavenge plate
5. Support ring with discharge aids
6. Spider ring with deflector plate
7. Thrust collar
8. Compression flange
9. Drive shaft with bearing housing and hydraulic motor
10. Protector sleeve
11. Seal arrangement (bearing housing)
12. Seal arrangement (distribution housing)
13. Upper bearing housing
14. Seal flush system
15. Pump unit for hydrostatic drive or other drives
16. Feed inlet
17. Filtrate outlet
18. Scavenge filtrate outlet
19. Heel drainage
20. Cake discharge
The operating advantages of the Seitz-Schenk Centrifugal Discharge Filter is the use of horizontal filter elements and the ability of automatic cake discharge without having to open the filter as described by Pall.

The Pall Seitz-Schenk Centrifugal Discharge Filter Type ZHF consists mainly of a pressure vessel with a hollow center shaft around which series of round filter elements are vertically stacked at specific, but variable, spacing. The filter stack, consisting of both the hollow shaft and the elements, is installed in the vessel, so that it can freely rotate. To clean the filter, the whole stack is spun by means of a drive system. The hollow shaft that serves as a filtrate discharge manifold is connected to an external drive motor, permitting the removal of cake by centrifugal action.

Funda filter (marketed in the United States by Steri Technologies Horizontal) is a centrifugal discharge filter where top-surface filter plates may be mounted on a hollow motor-connected shaft that serves both as a filtrate discharge manifold and as a drive shaft to permit centrifugal removal of the cake.

The Funda filter is driven from the top, leaving the bottom unobstructed for inlet and drainage lines; however, the Schenk filter employs a bottom drive, providing a lower center of mass and ground-level access to the drive system (Perry, 1999).

### 3.6.6.4 Air Filters: Tube Filters or Liquid Bag Filters

Air filters are used quite extensively to remove suspended dust or particles from air streams. The air or gas moves through a fabric and the dust is left behind. These filters are particularly useful for the removal of fine particles. One type of bag filter consists of a number of vertical cylindrical cloth bags 15–30 cm in diameter, the air passing through the bags in parallel. Air bearing the dust enters the bags, usually at the bottom and the air passes out through the cloth. A well-known example is a bag filter for dust found in the vacuum cleaner. Some designs of bag filters provide for the mechanical removal of the accumulated dust. For removal of particles less than 5 μm diameter in modern air sterilization units, paper filters and packed tubular filters are used. These cover the range of sizes of bacterial cells and spores.

The advantages of the tubular filter are that it uses an easily replaced filter medium, its filtration cycle can be interrupted and the shell can be emptied of prefilter at any time without loss of the cake, the cake is readily recoverable in dry form, and the inside of the filter is conveniently accessible.

A cylindrical filter bag sealed at one end is inserted into the perforated tube. The open end of the filter bag generally has a flange or special seal ring to prevent leakage. Slurry under pressure is admitted to the chamber between the head of the shell and the tube sheet, and then enters and fills the tubes. Filtration occurs as the filtrate passes radially outward through the filter medium and the wall of each tube into the shell and on out the filtrate discharge line, depositing cake on the medium. The filtration cycle is ended when the tubes have filled with cake or when the media have become plugged (Perry, 1999).

### REFERENCES


Butters, J. R. 1993. Notes on Separation Operations. Department of Food Science and Technology, University of Reading, UK.


