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Membrane Separation Process in Wastewater Treatment of Food Industry

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1. Introduction

Wastewater derived from food production is highly variable, depending on the specific types of food processing operations (e.g., fruit, vegetable, oils, dairy, meat, and fish). Advances in membranes technology have showed many advantages for wastewater treatment of food industry. By implementing membranes, the separated substances and clean water are often recoverable in a chemically unchanged form and are therefore easily re-used. Maximum benefits are obtained when one or both the output streams from the membrane system are recycled or re-used, thereby reducing process materials requirement and minimizing waste disposal costs.

This chapter reviews the development and applications of membrane processes in wastewater treatment of food industry. Particular focus is given to membrane’s special abilities to wastewater treatment for water regeneration and various re-uses purposes. Influence of engineering aspects is analyzed, specially operating conditions near critical flux to improve processes in wastewater treatment. Detailed discussions are provided with respect to constituents of concern in water reuse applications including recovery of other products with value for food industry.

2. Wastewater of food industry volume and quality

The types of food production processes (e.g., fruit, vegetable, oils, dairy, meat, fish, etc.) vary widely, with associated differences in the specific wastewater contaminants. The characteristics and generation rates of food wastewater are highly variable, depending on the specific types of food processing operations, including wastewater from of activities of food cleaning (sanitizing, peeling, cooking, and cooling); mechanically activities (conveyor medium to transport food materials throughout the process) and clean production equipment between operations. In addition, one important attribute is the general scale of the operations, since food processing extends from small, local operations.

Food processing can be divided into four major sectors: Meat, poultry and seafood; fruit and vegetables; dairy and beverage. Table 1 shows the wastewater volume and pollution charge of some food industries.
Primary and secondary treatments are often used to decompose the high organic contents of wastewater of food industry by aerobic and anaerobic fermentation processes. After of traditional treatment of wastewater, general requirements are covered by regulations of each country, usually complemented by consent limits based on avoidance of pollution. Discharge licenses may include maxima for flow, temperature, suspended solids, dissolved solids, BOD₅, nitrogen, phosphorous and turbidity. According at quality of water, in most cases, final disposal of treated waste water is into a water course where it will be diluted by the existing flow. However, subsequently one advanced process of effluent treating can be an option desirable to recycle water within a factory of food processing.

### 3. Membrane process

Membrane filtration is a process used to separate dissolved substances and fine particles from solutions. Membrane acts as a semipermeable and selective barrier that separates particles based on molecular or physical size. Solutes smaller of solution than the membrane pore size are able to pass through the membrane as permeate flux while particles and
molecules larger than the membrane pore size are retained. The two fluxes at outlet of membrane are important because this process has a high efficiency in the separation. The majority of commercial membranes are made usually of organic polymers (polysulfones and polyamides) and inorganic materials (ceramic membranes based on oxides of zirconium, titanium, silicium and aluminum).

The membranes are implemented in several types of modules. The membrane configuration determines the manner in which the membrane is packed inside the modules. Four main types of membrane configurations are used in the industry. These are: plate-and-frame, spiral wound, tubular and hollow-fiber configurations. The membrane geometry is planar in the first two and cylindrical in the two others. Figure 1 shows schematically a typical hollow fiber module (Okokchina, 2010).

![Figure 1. Scheme of the hollow fiber membrane module with crossflow. A large surface/volume ratio is expected for these modules.](image)

The membrane system is operated in a cross-flow feed mode. The concentrated stream passes parallel to the membrane surface as opposed to perpendicular flow that is used traditionally in filtration. This operating mode allows that accumulation of solute molecules at the membrane surface decreases and the permeate flux remains constant for a long time due to decreased hydrodynamic resistance at the membrane surface by cross-flow induced hydraulic turbulence. Flow direction is usually inside-out, i.e. the concentrate flux inside the fibers and the permeate flux is collected at the shell-side. It is often possible to reverse the flow (outside-in) for cleaning and unclogging of the membrane. Cylindrical configuration provides the possibility of maintaining high tangential velocity in the feed stream and is therefore particularly suitable for applications where the feed contains a high proportion of suspended solids or must be strongly concentrated.

The choice for a certain kind of membrane system is determined by a great number of aspects, such as costs, risks of plugging of the membranes, packing density and cleaning opportunities. The effects of the feed properties, the membrane properties, and the filtration conditions are obviously very important for the success of a membrane filtration process. Principal limitation of membrane lies in membrane fouling which is mainly associated with the deposition of a biosolids cake layer onto the membrane surface (McCUTCHEON & ELIMELECH, 2006; MI & ELIMELECH, 2008). However, several alternatives have been implemented to enhance this problem (AI-AKOUM et al., 2002; JAFFRIN et al., 2004).

### 3.1 Membrane applications in food industry for wastewater treatment

Membrane separation process has special recognition in food wastewater treatment, applied to the end of conventional treatment systems (VOUCH et al., 2008). The process is used...
primarily to reduce the volume of the food wastewater that is achieved by recovering of two fluxes: permeate water flux having the majority of the original volume, and concentrated flux in a lesser volume (constituents of effluents retained).

The membranes used in food wastewater treatment differ widely in their structure and function. Mainly they are operated in four membrane processes: microfiltration (MF), ultrafiltration (UF), nanofiltration (NF) and reverse osmosis (RO). Solvent permeability and separation selectivity are the two main factors characterizing at these membranes. Transport mechanisms and operating membrane conditions can also explain the pass of species through membranes. Particle size is practically the sole criterion for describer the permeation or rejection of membranes. However, microporous membranes (NF and RO) have ability of separate particles at molecular level and their selectivity is mainly based on the chemical nature of the species.

Several works have been focused on these factors to explain separation selective of residues of food wastewater. Effluents treatment of dairy industry by RO and NF membranes are reported in many investigations, however, a strong development and growth of membrane technology can be observed in the results from the other food industries (Turano et al., 2002). Food industry standards specify that, spent process water intended for reuse (even for cleaning purposes) must be at least of drinking quality. Regulations for other applications, such as boiler make-up water or warm cleaning water, are even more stringent. There has been a study on the possibilities for reuse of vapor condensate in a milk processing company (dried milk production) as boiler make-up water (Hafez et al., 2007), and the reuse of chiller shower water in a meat processing company (sausage production) as warm cleaning water (Mavrov & Bélières, 2000).

### 3.2 Membrane characteristics

Generally membranes are characterized by pore flow or molecular weight of particle that is retained or is filtered by the membrane. However, important membrane properties such as structure, porosity, thickness, wettability surface and operating conditions, are also studied because affect rejection of solutes. The electrostatic repulsion between the membrane surface and the contaminant may be particularly analyzed to enhance waste solute retention and to increase water flux.

The smallest particle size present in the feed is very important for the selection of membrane pore size. However, currently the feed properties can be changed by pretreatments such as pH adjustment, thermal treatment, addition of chemicals, and pre-filtration. The pH adjustment (Luo et al., 2010) and thermal treatment can decrease the precipitation of certain substances. In addition, chemicals can be added to the feed to increase the particle size through aggregation, and the retention of specific substances can be enhanced through micellation or complexation (Wu et al., 2007). The salt concentration of the feed and the valence of the salt present can also be important to select membrane type (Muro et al., 2009; Lefebvre & Moletta, 2006).

#### 3.2.1 Pore-flow and material membranes

Membrane pore flow is differentiated by the size of particles diameter that they can separate (micrometers, µm) and nominal molecular weight cutoff MWCO (kilo Daltons), which is a performance-related parameter, defined as the lower limit of a solute molecular weight for which the rejection is 95-98% (Boerlage et al., 2004). In theory, compounds having a molecular weight greater than the molecular weight cut off (MWCO) will be retained by the
Membrane and compounds with molecular weights less than the MWCO will pass through the membrane as permeate. Table 3 shows size range of particles retained with range of MWCO membranes for treatment of wastewater of food industry.

| Membrane Process | MWCO membrane (kilo Daltons range) | Retained diameters particle (µm range) | Retained solutes | Application in effluents treatment of food industry |
|------------------|-------------------------------------|----------------------------------------|------------------|--------------------------------------------------|
| MF               | 100-500                             | $10^{-4}$-$10^{-1}$                     | Bacteria, fat, oil, grease, colloids, organics microparticles | Oil, Cereal, Dairy, Beverage                       |
| UF               | 20-150                              | $10^{-3}$ – $1$                         | Proteins, pigments, oils, sugar, organics microparticles     | Dairy, Cereal, Oil, Tomato puree, Beer, Wine, Fish, Meat, Pickled vegetables |
| NF               | 2-20                                | $10^{-2}$ – $10^{-1}$                   | Pigments, sulfates, divalent cations, divalent anions, lactose, sucrose, sodium chloride | Olive oil, Dairy, Beverage, Meat canning, Pickled vegetable |
| RO               | 0.2-2                               | $10^{-4}$ – $10^{-3}$                   | Salts, sodium chloride and inorganic ions                    | Dairy, Cereal, Fish, Meat, Pickled vegetables      |

Table 3. Typical range of application of MWCO, diameter particle and retained solutes type by membrane process in wastewater treatment of food industry.

Retention is obviously affected by the pore size due to the sieving effect, especially when using MF and UF membranes. With tighter (NF and RO) membranes retention will be governed more and more by the electrostatic forces as well as by other interactions between membranes and solutes. Thus MWCO is only a rough indication of the membrane’s ability to remove a given compound as molecular shape because polarity and interaction with the membrane affect rejection (Guizard & Amblard, 2009).

Respect to pore diameter, it has frequently been seen that the membrane with the most open pores does not usually give the highest permeate flux in filtration process. Porosity (ratio of void space to total membrane volume in porous membrane) and pore size distribution may influence the apparent size of particles retained. Typical microporous membranes have average porosities in the range 30%–70%. Porosity can also be measured by analyzing processed images obtained from microscopic analyses such as scanning electron microscopy (SEM). Figure 2 shows SEM image of an asymmetric porous structure of a ceramic membrane. It may be noted that the membrane has fine pores through which raw water is filtered (Figure 2a). The most of ceramic membrane elements are constructed from supported multiple ceramic layers constituting an asymmetric porous structure. Carbon macroporous material is used as support for ceramic membrane deposition (Figure 2b and 2c). Multiple layers are usually resulting from residual spaces created between ceramic particles during sintering. The bottleneck geometry is representative of pores resulting from sintering of almost spherical particles, for example, this is the case of porous structures obtained with titania, zirconia (Guizard et al., 2002; Guizard & Amblard, 2009). The porous sites are uniformly distributed in the membrane and effective diameter of the membrane pore can be determined assuming pores are circular in shape. However, pore geometry (tortuosity; $\tau$) can also affect the retention of molecules by a membrane. Tortuosity reflects the length of the average pore compared to the membrane thickness. Cylindrical
pores at right angles to the membrane surface have a tortuosity of one, that is, the average length of the pore is the membrane thickness (Cho et al., 2000; Zhao et al., 2000; Vrijenhoek et al., 2001).

Fig. 2. SEM image of cross section of a ceramic membrane porous structure of MF, with cut off of 300 kDa and 5 µm pore size, used in wastewater treatment of food industry (From Escobar. PhD thesis, Institute Technological of Toluca, Mexico, 2010)

Chemical composition, hydrophilicity/hydrophobicity, charge, and morphology have also significantly effect on permeability and stability of the membrane (Khayet et al., 2005). Particularly, ceramic membranes have a composite structure, which is used to increase the permeability for small pore size membranes by decreasing the overall hydraulic resistance (Peng et al., 2005; Yu et al., 2006) while polymeric membranes can be modified to make them more hydrophilic and achieve less fouling and better cleaning efficiency.

3.2.2 Surface pore charge. Isoelectric point
Membrane charge affects membrane efficiency in food wastewater treatment, particularly when low cutoff membranes are used for treatment effluents with high salts concentration. The charging occurs due to, for instance, dissociation of functional groups, adsorption of ions from solution, and adsorption of polyelectrolytes, ionic surfactants, and charged macromolecules. Generally, membrane materials carry a negative charge or are modified to have a negative charge because natural organic matter in water is negatively charged at neutral pH, due to phenolic and carboxylic functional groups (Kaeselev et al., 2002). A negatively charge membrane, therefore, prevents rapid deposition of foulants on the membrane surface by charge repellence An increase in the flux of a relatively dense membrane at a high pH may result from an increase in membrane hydrophilicity due to the dissociation of the functional groups in the membrane structure (Schaep & Vandecasteele, 2001; Zhao et al., 2005). Many polymeric membranes are amphoteric, having both negatively and positively charged functional groups in the polymer matrix. Ceramic membranes can also show in water amphoteric behavior and thus their surface charge is pH dependent (Cho et al., 2000).

Membrane charge, as well as hydrophilicity property, can be predicted based on known membrane chemical structure. However, membrane surface/pore charge can be measure by electrical potential (Martin et al., 2003). When the membrane contains strongly acidic groups, the dissociation of the groups occurs immediately at a low pH, and the zeta
Potential can be expected to be strongly negative even at low pH values (pH 2–3); while when the membrane contains weakly acidic groups, the zeta potential can be expected to become more negative from the point the groups start to dissociate to the point where the groups are totally dissociated. Similarly, strongly basic groups give positive potentials in most of the pH range, while weakly basic groups have no positive charges at pH values higher than 8 (Kim et al., 2005).

The isoelectric point (IP) (pH where net charge is zero) of a membrane is also a referent to determine the behavior of their surface charge, depending on the pH of the wastewater in contact with the membrane. (Cheng et al., 2008). For example, typically NF polimeric membranes are negatively charged at neutral pH, with IP around pH 3-4, while ceramic membranes have a IP around pH 6-7.

The IP of a membrane can be evaluated from the pH dependence of the zeta potential (Martin et al., 2003). However other experiments can also describe this parameter. Figure 3 shows isoelectric point of a ceramic membrane of zirconium and titanium oxide.

Fig. 3. Isoelectric point of a ceramic membrane of UF. (a) pH of permeate water is measured during an operation time range. (b) pH differential is determinate when pH feed solution is adjustment at 4-8 range, intersection of line with horizontal axe denotes isoelectric point at 6.2. (c) The values of zeta potential are measured in dependence with pH of ionic solution.

Figure 3a, denotes pH determination of pure water during water filtration by membrane of UF. In this membrane the pH value of permeate does not change with operation time. It shows that isoelectric point is around pH 6. Figure 3b shows the pH differential permeate water dependent of pH feed solution. Intersection of line with horizontal axe denotes isoelectric point at pH 6.2 for the same ceramic membrane. Figure 3c shows the values of zeta potential and pH of ionic solution; the values were measured through the pores of ceramic membrane. Intersection line with pH axis, indicates that isoelectric point is 6.2.

4. Influence of engineering aspects in food industrial wastewater treatment

Systematic studies of membrane applications in food wastewater treatment are focused on membrane functionality and performance filtration, under different operation condition. Several researches are specifically focused on optimizing crossflow hydrodynamics and/or membrane filter geometry to increment performance of water flux and maximum rejection or recovering species from effluents. Hydrodynamic factors affecting the membrane functionality, are cross-flow velocity ($v$) and transmembrane pressure (TMP). Permeate
flux can increase or decrease due to simultaneous influence of these variables. Temperature, dilution and pH are also variables involved in the membrane efficiency in membranes filtration. Permeate flux increases with increasing feed temperature due to a decrease in viscosity and/or due to an increase in solubility of suspended solids (Galambos et al., 2004). The exception is the presence of calcium and magnesium salts that might precipitate when temperature is increased. This problem can be avoided at least in some cases through feed pretreatment (Sarkar et al., 2006). The pH has a significant influence on the permeation rate especially around the isoelectric point of certain colloids where they tend to destabilize and precipitate. It also has an effect because of the changes in surface charge of the membrane either due to the amphoteric nature of the surface or due to the specific adsorption of species as presented earlier (Vourch et al., 2008).

4.1 Cross-flow velocity
A hydrodynamic variable of membranes in cross-flow filtration systems is essentially the velocity at which the feed flow is passed across the surface of the membrane. Crossflow velocity (ν) is linear velocity (m/s⁻¹) of the feed flow circulating tangentially across the membrane. This parameter is described by relation of feed flow rate (Qw; m³/s⁻¹) and the cross sectional area of feed membrane (As; m²).

Turbulent flow conditions are recommended to maintain the flow tangential to the membrane, thereby reducing the phenomenon of concentration polarization and, consequently, the accumulation of solute near the membrane and inducing acceptable permeate flux for long time. Shear effects induce hydrodynamic filtration of the particles from the boundary layer back into the bulk, with a positive effect on the permeate flux. However, as feed concentration increases, it becomes more difficult to maintain a high recirculation velocity due to an increase in feed viscosity (Muro et al., 2009). In addition, if foods waste water containing macromolecular solutions with flexible solutes, thus also a high velocity can cause deformation of the polymer chains, which favors certain macromolecules that pass through the pores.

The hydrodynamics flow can also be characterized by calculating the Reynolds (Re) number by equation (1).

\[ Re = \frac{\nu d_h}{\mu} \]  

Where ν is crossflow velocity, dh hydraulic diameter of membrane module and μ the dynamic viscosity of fluid.

Normally, Re>2100 guarantees a turbulent flow in the module and a minimum thickness for the concentration polarization layer. Prevention of reversible fouling layer formation is sufficiently achieved by a crossflow velocity of around 2.0 ms⁻¹ in UF membranes (McKeown et al., 2005). In practical applications, one has to keep in mind that the permeate flux will be determined by the combination of crossflow velocity and TMP (See Figure 5).

4.2 Transmembrane pressure
The driving force for transport behind membrane process MF, UF, NF and RO, is the pressure difference between feed and permeate flux of the membrane (TMP; bar, psi). TMP is defined as the difference in pressure between the filtrate side of the membrane and the permeate side of the membrane. The average TMP is in general calculated as follows:
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\[ \text{TMP} = \frac{P_i + P_o}{2} - P_p \]  

(2)

Where \( P_i \) is pressure at the inlet of the membrane module; \( P_o \) is pressure at the outlet of the membrane module and \( P_p \) is permeate pressure.

The permeate flux depends directly on the applied TMP for a given surface area under uniform operational conditions. The flux of the pure water is linearly pressure dependent. However when food wastewater is treated by membrane system the flux is more complex. The behavior depends of wastewater composition, membrane type and crossflow velocity.

In food wastewater treatment, one has to keep in mind that the permeate flux will be determined by the combination of crossflow velocity and TMP, due to contaminants (Sarkar et al., 2006; Blöcher et al., 2002; Oktay et al., 2007; Avula et al., 2009).

Figure 4a and 4b show the effect of crossflow velocity and TMP on permeate flux using two membranes of different MWCO (300 kDa and 15 kDa). The experiments were performed by Escobar, 2010. The results indicated that the flux enhancement caused by increasing crossflow velocity was particularly pronounced at range values of the TMP (3-5 bar) and crossflow velocity of 3 ms\(^{-1}\). Fouling occurred over a range of TMPs of 5-6 bar and crossflow velocities at 3.5 ms\(^{-1}\). The permeate flux decreased with time during the development of the fouling layer, but once the fouling layer was established, the permeate flux became constant for a given set of experimental conditions. Therefore these results indicate that at moderate values of TMPs and high flow rates at the membrane surface are operating conditions that conducive at high permeate fluxes in these experiments. Besides, figure 4c shows an overall positive effect of enhanced flow hydrodynamic conditions (TMP = 4 bar) on the average permeate flux, although in the turbulent regime (Re>3,000) a weaker correlation and more data scattering were observed. Therefore a clear correlation between the 3 h flux and Re in the transient regime (Re<3000) could be expected.

Fig. 4. Effect of crossflow velocity and TMP on the 3 h permeate flux in wastewater treatment of a cereal industry using membranes of MF and UF (a) 300 kDa. (b) 15 kDa. (c) The interdependence between average flux and hydrodynamic conditions for two membranes in a wide range of Re numbers at TMP = 4 bar (From Escobar 2010. PhD thesis, Institute Technogical of Toluca).

Particularly, operational membrane conditions in wastewater treatment show moderate TMP and high flow rates at the membrane surface are conducive of high permeate fluxes in
the MF and UF. An increase in TMP is required to maintain a particular water flux (constant-flux operation) independently of the membrane type and MWCO. However, an increasing flux could lead to an increase in polarization and fouling, which will limit the permeate flux (Abbasi et al., 2011; Simate et al., 2011).

High pressure can also allow membrane compaction, ultimately resulting in the formation of a denser membrane with smaller pores, or one possible enlargement of membrane pores with time, which enables particles to penetrate through the membrane matrix. Choi et al., (2005) showed clearly that pore sizes are modified in the membrane matrix increased with increasing TMP.

### 4.3 Permeate flow rate

The functionality of a membrane in wastewater treatment is determined by water permeation capacity and retention of solutes. Although permeate flux depends of the characteristics of the membrane and quality of wastewater, the average pore size and pore-size distribution is important since it will give an indication of which transport mechanism can be expected to be dominant for a given specie mixture in a defined material and at given process conditions. There are two theory models to describe the mechanism of permeation in membrane process; one is the solution-diffusion model, in which permeants are diffused through the membrane down a concentration gradient. The other model is the pore-flow model, in which permeants are transported by pressure-driven convective flow through tiny pores. Separation in this case, occurs by excluding of some particles of the pores in the membrane. Fick’s law describes the mass flux through an area perpendicular to the flow direction (Miyoshi, 1998):

\[
\frac{dV_i}{dt} = J_{pi} = -D_i K_i \frac{dC_i}{dx}
\]  

(3)

Where $J_{pi}$ is the linear fluid velocity (ms$^{-1}$) of component (i) or permeability flux (Lm$^{-2}$h$^{-1}$). The diffusion coefficient $D_i$ (ms$^{-1}$) reflects the mobility of individual molecules in membrane material and the molecule sorption coefficient $K_i$ reflects the number of molecules dissolved in the membrane material. The product $D_i K_i$ is membrane permeability and is a measure of the membrane’s ability to permeate species. $dC_i/dx$ is the concentration gradient (molL$^{-1}$) for component (i) over the length x (m). $V_i$ is the volume of substance (i) transferred (L), t is time (h) and A is perpendicular area (m$^2$).

Permeability flux $J_{pi} = V_i / At$ is obtained by equation integration (3) and applied for $dx = x$ (membrane thickness or membrane resistance for the pure water transport). $C_{i0}$ and $C_i$ are the concentration of component (i) on the feed side and concentration of component (i) on the permeate side respectively. Solution-diffusion model is often used to describe the transport in RO membranes.

\[
\frac{V_i}{At} = -L_p \frac{\Delta P}{x}
\]

(4)

$L_p$ is the hydraulic permeability coefficient (Lm$^2$bar h$^{-1}$); $\Delta P$ is gradient pressure TMP (bar) in membrane system. Information about porous structure and viscosity of the filtrated liquid is contained in $L_p$ factor.

Membrane resistance (x) is a measure of the hydraulic resistance to flow through a pore channel. However, when wastewater is fed, increment of TMP can cause a decreasing of
membrane permeability because of hydraulic resistance increment by the fouling phenomena. Increment of crossflow velocity, dilution of wastewater, change of temperature of feed and using turbulence promoters such as backflow techniques, feed pulsation and rotation of filter elements, are hydrodynamic methods to increment permeate flux and reduce the hydraulic resistance due to fouling (Jaffrin et al., 2004; Luo et al., 2010).

4.4 Selectivity factor
The best measure of the ability of a membrane to separate molecules (i) of wastewater, is the ratio of their permeability \( \alpha_i \), called the membrane selectivity, which can be written in terms of the apparent sieving coefficient:

\[
\alpha_i = \frac{C_{ip}}{C_{if}}
\]  

(5)

\( C_{ip} \) is concentration of specie (i) in the permeate flux and \( C_{if} \) is the concentration of specie (i) in the feed flow.

The selectivity of a membrane depends on its ability to transmit different species to different extents. Factors that affect solute transmission are solute type, membrane type, solution pH, solution ionic strength, the permeate flux, and the hydrodynamic conditions on the feed side. Membrane selectivity is most often expressed as the membrane retention, \( R \), toward the species to be separated. \( R \) is dimensionless parameter, with variation range of 0-100 %.

\[
R = \frac{C_{if} - C_{ip}}{C_{if}} = (1 - \alpha_i)
\]  

(6)

| Membrane/Cut off (kDa) | Ions concentration (mgL\(^{-1}\)) in water permeate |
|------------------------|---------------------------------------------------|
|                        | Na\(^{+}\) | K\(^{+}\) | Ca\(^{2+}\) | Fe\(^{3+}\) |
| MF/150                 | 4        | 5        | 4        | 5        | 4        | 5        | 4        | 5        |
|                         | 140      | 148      | 23.0     | 25.1     | 16.2     | 16.2     | 6.4      | 9.9      |
| UF/15                  | 133      | 135      | 22.2     | 20.1     | 13.1     | 13.0     | 5.7      | 2.1      |

Table 4. Effect of (TMP) on the permeability of some ions by MF y UF membranes

Rejection of neutral organic solutes generally increases with the molecular weight (or diameter) of the solute. Species will be retained by the membrane according to their size (sieving effect). For a mixture of multivalent and monovalent co-ions in the feed, multivalent co-ions are retained due to their higher electrical charge, while a part of monovalent co-ions pass through the membrane with counter ions to fulfill charge equilibrium criterion on both sides of the membrane (Lefebvre et al., 2003). However, the absolute values of the salt rejection vary over a wide range; the ranking for the different salts is the same for all membranes (Rautenbach & Albrecht, 1989). A high TMP value also affects the selectivity of some ions species. Table 4 shows the effect of TMP conditions on permeability of some ions by two ceramic membranes (Muro et al., 2009). Ions were identified in wastewater of a food industry. The experiments were performed to determine...
the effect of pressure increment on selectivity of these membranes for these ions. The results indicate that for all PTM values, the ions Fe$^{3+}$ and Ca$^{2+}$ were slightly declined, while ions Na$^+$ and K$^+$, were filtered by both membranes.

For other hand, exceptional selectivity for a number of important separations in wastewater treatment of food industry are mentioned in several reports (Vourch et al., 2008; Muro et al., 2010; Escobar et al., 2011; Simate et al., 2011).

Figure 4a and 4b show the difference between selectivity of two ceramic membranes of MF (300 and 150 kDa) and one of UF (50 and 15 kDa) for various TMP values. The data were obtained by experimental study of organics species in micelles with two colorants (a) Brilliant blue. (b) Tartrazine. Membranes denote a low selectivity for the colorants and a high permeability for water. Particularly, membrane of 15 kDa shows the lowest selectivity for two colorants for all TMP values. SEM image denotes, particles deposited on membrane surface, showing a low selectivity of a membrane of 300 kDa for tartrazine colorant.

5. Critical flux conditions

During membrane filtration process are identified three regimens in accordance to the critical flux theory (Field, 1995). Figure 6 shows a typical flux profile by three membranes.

Subcritical regime is the first stage of filtration, where flux varies linearly and reversibly with TMP, a high crossflow velocity is employed to increase capacity of permeation and a critical pressure is achieved in the end of this regime Processes where high water purity is required are carried out regime I, because membrane selectivity is optimal. The flux in regime II is independent of TMP, which can be described by an equilibrium stage, where the transport of particles toward the membrane is balanced with the transport of particles toward the bulk flow. At high TMP values, the permeate flux is not significantly affected by increases in pressure. This limiting flux or critical flux increases with increasing crossflow velocity, because materials deposited on the membrane by mass transport are removed by

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the wall shear force. For soluble species and fine colloids, the critical flux can be considered as the flux below which the wall concentration does not initiate fouling (Cho & Fane, 2000). High capacity of the concentration of species from wastewater can also be achieved in this regime and the critical flux may either be identical to the clean water flux at the same TMP (Hwang et al., 2006). However, outside the limiting flux, operation at sustained permeability and selectivity is not possible due to the accumulation and compaction of the fouling layer on the membrane. Finally flux decline in time-dependent with high pressure above the critical TMP, are identified in regime III due to increment membrane fouling. Their removal is necessary for stable membrane operation (Espinasse et al., 2002).

![Critical flux regimes in membranes of 300, 150 and 15 kDa: (I) Subcritical operation (II) Critical operation (III) Decline flux.](image)

The critical flux value depends largely on the hydrodynamic conditions in the process, the membrane pore size, and the feed physicochemical condition (Mänttäri & Nyström, 2000). Appropriate manipulation of these parameters, specifically the hydrodynamic condition, may lead to increment of flux and the reduction or even the elimination of both reversible and irreversible fouling of the membrane. The critical flux can be experimentally identified through constant flux filtration experiments by incrementing the flux until the TMP is no longer steady.

6. Membrane fouling control in food industry for wastewater treatment

Fouling is the most important issue affecting the development of membrane filtration as it worsens membrane performance and shortens membrane life (Boerlage et al., 2004). Membrane fouling by food wastewater filtration is attributed to deposition of species from effluents onto the membrane surface or within membrane porous, it causes a permeate flux decline with time because the filtration resistance is significant increased (Foley, 2006). Fouling studies on membranes are based in proteins deposition and their interaction in membranes surface. Polydispersity of naturally occurring macromolecules such as polysaccharides and humic substances, have also added a particular complexity on investigation to the fouling membrane mechanisms. Advances in understanding fouling of
other species such as bacteria, yeast, emulsions, suspensions, salts and colloids from food wastewater have occurred in microfiltration and ultrafiltration literature (Chan et al., 2002; Foley et al., 2005; Hughes & Field, 2006; Cheng et al., 2008).

There are two forms of membrane fouling: the fouling layer that is readily removable from the membrane, it is often classified as polarization phenomena or reversible fouling and is removed by physical procedures. Internal fouling caused by adsorption of dissolved matter into the membrane pores and pore blocking is considered irreversible, which can be removed by chemical cleaning and other methods (Hughes & Field, 2006).

Several aspects such as pretreatment of feed solution (example add flocculants before filtration), membrane surface modification, operating conditions and heavy cleaning procedures such as high temperature, while using caustic, chlorine, hydrogen peroxide, ozone, and strong inorganic acids are carried out on the membrane plant in operation to decrement fouling problem. Hydrodynamic methods used for performance enhancement of membrane filtration as back-pulsed (permeate flow reversal technique), creation of pulsed flow in membrane module, TMP pulsing, creation of oscillatory flow, generation of Dean vortices in membrane module, generation of Taylor vortices in membrane module and use of gas-sparging, have also been developed to reduce membranes fouling (Parck, 2002; Choi et al., 2005; Luo et al., 2010). Specifically, rapid accumulation of foulants, is usually referred to the critical flux (Chan et al., 2002). For single particles deposition, the critical flux occurs at a particular hydrodynamic condition (Espinasse et al., 2002). Critical flux condition can be determined by adsorption process, a slow increase in membrane resistance is always detected by the kinetics of this adsorption, particularly for proteins (Hughes & Field, 2006; Vyas et al., 2002; Ognier et al., 2002). For complex fluid systems, one common practice to experimentally determine the critical flux value is to incrementally increase the flux for a fixed duration. This leads to relatively stable TMP at low fluxes (indicating little fouling), and an ever increasing rate of TMP rises at fluxes beyond the critical flux values (Knutsen and Davis, 2006). In fluids with both macromolecules and particulates, membrane fouling takes place even at low flux rates, but changes dramatically when critical flux is reached. Although rigorous mathematical expressions to determine membrane fouling, have been reported (Rögener et al., 2002b; Lefebvre et al., 2003), experimental critical flux determination remains an efficient approach to assess the fouling behavior of a given filtration system and to compare different operating conditions (Clech et al., 2006).

7. Optimization membrane process in food industry for wastewater treatment

In order to use membranes filtration as an efficient separation technique and economically interesting, the process optimization is essential. The purpose of the optimization process is the achievement of the highest possible flux production for a long period of time, with acceptable pollution levels.

A well chosen wastewater pretreatment and a proper selection of membrane in relation to the species properties from effluents can be used to assess and predict the optimal flux during filtration. However, the control of the feed pH, ionic strength and temperature is often necessary in order to maximize removal of food production residues.

Optimization methods and statistical designs are widely employed in various field of science from chemistry to engineering to enhance the membrane processes. Particularly, Response Surface Methodology (RSM) is a sequential form of experimentation used to help
predict or optimize process. The variables are integrated in a mathematical-statistical model to express the possible simultaneous influence of membrane characteristics, fed composition and operating conditions on water flux performance. Several membrane processes and operating conditions have been reported in the treatment of food wastewater (Stoller and Chianese, 2006; Iaquinta et al., 2009; Escobar et al., 2011).

Table 5 summarize some results that describe the treatment wastewater optimization from production of these food. The permeate water fluxes are different in optimization process, due to membrane type used, membrane area and fed wastewater quality.

| Reference              | Food wastewater | Membrane process/membrane area (m²) | Optimum conditions                                                                 | Maximal permeate flux (L/h m⁻²) |
|------------------------|-----------------|-------------------------------------|-------------------------------------------------------------------------------------|---------------------------------|
| Stoller and Chianese,  | Olive oil       | UF/32 NF/32                         | Oil concentration, feed flow velocity, temperature, critical flux, membrane type      | 415.8                           |
| (2006)                 |                 |                                     |                                                                                     | 222.0                           |
| Iaquinta et al., (2009) | Tomato puree    | NF/2.51                             | Fed concentration, conductivity, temperature, , feed flow velocity, transmembrane pressure | 8.21                            |
| Escobar et al. (2011)  | Cereal          | UF/0.56                             | Transmembrane pressure, membrane type, dye concentration (brilliant blue and tartrazine), flow velocity, filtration time | 19.5                            |

Table 5. Membrane conditions in treatment food wastewater optimization

8. Recovery of food industrial effluents by membrane process and water reuse

The drivers for implementation of water reuse practices in food industries is essential due to increasing demands on declining freshwater supplies, severe water shortages and dry periods, and the fact that water quality discharge regulations have become stricter. In addition, environmental and economical aspects are incentives to treat food wastewater with water reuse purpose (Casani et al., 2005).

Food industry looks at membrane processes for wastewater treatments to produce purified water for recycle or reuse due to their characteristics as techniques that can be implemented in any food plant and because they can be combined with other unit operations (hybrid processes (Sarkar et al, 2006). Table 6 summarizes some important results of recycling water and cleaning effluents by membrane technology.

Typical wastewaters in food industries come from different parts of the plant and they are submitted to a wide fluctuation in flow and composition depending on the type of food industry and size and even, on the moment in which the plant is working (different steps of “cleaning in place”, heating, sterilization, etc.). They do not contain toxic compounds (except in wastewater from washing fruits and vegetables in which pesticides can be a water contaminant) but they are characterized by high values in biological oxygen demand (BOD) and chemical oxygen demand (COD) as well as total dissolved solids (TSS) in some cases. Those high contents come from organic (proteins, carbohydrates, fats) and inorganic (salts, additives, dyes) compounds.
| Reference | Industry/wastewater source | Combined membrane treatments | Water recycling |
|-----------|-----------------------------|------------------------------|-----------------|
| Chmiel et al., (2003) | Dairy/Vapor condensates from concentration and drying steps | Cartridge filtration-NF-RO-UV-oxidation | Drinking |
| Mavrov et al., (1997), (2000); Chmiel et al., (2000); Čuda et al., (2006); Vourch et al., (2008) | Combined membrane treatments | Two NF steps | Water use in boilers |
| Koo et al., (2011) | Dairy/Flash coolers | Cartridge filtration-NF-UV | Boiler make up water |
| Rögener et al., (2002a), (2002b), (2002c); Tay & Jeyaseelan, (1995) | Milk/Bottles machines, cheese processing | UF and RO | Unspecified |
| Scharnagl et al., (2000); Muro et al., (2010) | Beverage/bottle rinsing, brewing room, bright beer reservoir | MBR-NF, RO | Unspecified |
| Rajkumar et al., (2010); Muro et al., (2009) | Fruit and vegetable processing/rinsing beans, cereal processing | MF, UF, NF, RO | Rinsing beans |
| Iaquinta et al., (2006), (2009); Mänttäri & Nyström, (2000) | Tomato/cleaning, sorting and moving the processed | NF | Unspecified |
| Noronha et al. (2002); Bölcher et al., (2002) | Fruit juices/ bottle washing, fruit processing, juice production and cleaning of tanks, pipes | NF | Drinking |
| Turano et al. (2002) | Vegetable oil/olive mill, washing. | MF, UF, NF, RO | Drinking |
| Mohammadi & Esmaeelifar, (2004); Galambos et al., (2004); Akdemir & Ozer, (2009); Mantzavinos & Kalogerakis, (2005); Rajkumar et al., (2010) | Meat and seafood/ slaughterhouse fish and crustaceans and tuna cooking | Two NF steps-UV SBR, MBR, UF and RO in different combinations | Drinking |

Table 6. Promising applications of membranes in wastewater treatment of food industry
8.1 Recovery of cleaning-in-place solutions

Special attention can give at the recovery cleaning solutions from wastewater of food industries. A large amount of acids and alkalis in cleaning and sanitizing steps are used in dairy industry. The consumption of NaOH, HNO₃ and detergents/disinfectants in a dairy industry that processes 1.5 million liters of milk per day is around 3 500, 1 000 and 1 000 kg per day respectively (Fernández et al., 2010). More than 40% of the total pollution caused by a dairy industry comes from their cleaning in place units (Henk, 1993). Particularly, the cleaning in place (CIP) used in food industries consists in a number of steps that depends on the type of product, but the final waste streams collected from each of these stages are usually treated together and show COD values of 400-600 mgO₂.L⁻¹ (Daufin et al., 2001).

There are a number of works describing how to recover contaminated cleaning solutions by membranes (Choe et al., 2005; Fernández et al., 2010; Gésan-Guiziou et al., 2002, 2007; Merin et al., 2002; Räsänen et al., 2002). Dresch et al. (2001) pointed out the NF technology as a promising technique compared to decantation, centrifugation and microfiltration (0.1µm cut-off) for the regeneration of waste NaOH solutions from an industrial CIP system. However, Gésan-Guiziou et al. (2007) reported that MF could be more adequate operation based on that the surfactant contained in the spent detergent is only slightly rejected by the membrane and costs of MF operation are much lower (lower TMP) compared to UF and NF costs, in spite of that the COD permeate when using MF was much higher and its possible uses can be limited.

When using NaOH or HNO₃ solutions in alkaline and acid cleaning steps, their recovery in the permeate is not very difficult, because the rejection of these compounds on an ultrafiltration or even in a NF are very low, obtaining a permeate stream than can be reused in the CIP and being the rest of foulants retained by the membrane. However, when the cleaning agent is composed by other chemicals (antisalants, anionic/cationic detergents, antifoaming compounds, surfactants, etc.) their recovery in the permeate stream is not so evident (Wendler et al., 2002). The use of MF, UF or NF techniques depends on if surfactants want to be recovered in the permeate or in the concentrate streams. If surfactants are below their critical micelle concentration (CMC) they will not be retained by any of these techniques, but if they are above CMC, MF and UF techniques retain these components and the permeate stream will lose its cleaning properties. Some works based on NF processes with the aim of surfactants recovery in the permeate stream have been published in the last years (Boussu et al., 2007, Forstmeier et al., 2002; Kaya et al., 2006, 2009). In those cases permeate flux and surfactant rejection are strongly dependent on the membrane material (membrane isoelectric point - IEP) and feed conditions (pH, concentration, etc.) due to that NF processes are not only governed by steric reasons and charge interaction between solutes and membrane surface plays an important role in transmission and membrane selectivity.

Diluted caustic and acidic washing solutions (showing COD between 8 000 and 10 000 mgO₂.L⁻¹) can be recovered by NF membranes with molecular weight cut off (MWCO) between 150 and 300 Da. Permeate flow rates are moderate (between 7 and 12 Lh⁻¹m⁻²) at pressures around 0.9 MPa (Räsänen et al., 2002). NF shows robust performance for the recovery of caustic solutions when faced with large variations of solution composition, as it happens at industrial CIPs (Dresch et al., 1999; Gésan-Guiziou et al., 2002). In some published research, transmission of NaCl higher than 99% was measured when variable feed composition (COD between 100 and 11 000 mgO₂.L⁻¹) and suspended matter between 0.4 and 5.6 gL⁻¹ was nanofiltered with ceramic membranes of 1 000 MWCO obtaining high permeate flow rates (40–110 Lh⁻¹m⁻²) at 70°C and 0.4 MPa transmembrane pressure.
Regarding to the acidic detergents used in food industries CIPs, some results have been published (Novalic et al., 1998). Two HNO₃ spent solutions were investigated with NF. Higher COD cleaning solution of 18500 mgO₂.L⁻¹ was obtained after a cleaning step without previous alkaline step. The other solutions was lower in COD (1800 mg O₂.L⁻¹) and was obtained after a previous alkaline cleaning step. Two effluents were nanofiltered at 50 °C and 3.0 MPa and at maximum recovery rate of 75%.

In other studies, several salts (Ca(NO₃)₂ and (Mg(NO₃)₂) were analyzed in the cleaning solution. However low COD solution essayed was nanofiltered at a rate of 40 Lh⁻¹m⁻² and final COD was low (450 mgO₂.L⁻¹). Kaya et al. (2009) used NF (1 000 Da cut-off) to treat a detergent composed by anionic and nonionic surfactants, dyes and salts from a dishwasher detergent. Maximum fluxes (around 120 Lh⁻¹m⁻², 25 °C, 1.2 MPa) were obtained at pH of 5, near to the membrane IEP. However, surfactants have hydrophobic interactions with anionic dyes (tartrazine) what explains higher rejection than expected (Kartal & Akbas, 2005; Zahrim et al., 2011). Authors found also strong influence of temperature and pH on the flux decay along the experiments. Initial higher fluxes at higher temperatures (40°C) rapidly decay due to pores blocking by surfactant monomers and rejections reduces with temperatures due to an increase in solutes diffusion or expansion of membrane structure a higher temperatures (organic membranes).

For other hand, large dairy companies (food companies in general) are changing the conventional cleaning agents for those novel single-phase detergents. These new formulations are expensive but CIP steps are shorter and only have one or two steps (cleaning and disinfection). Single-phase detergents are designed by detergent companies and formulations are not available but alkalis or acids, surfactants, complexant agents and de-foamers usually are included. Recovery of these detergents is not easy because all the components should be permeate through the membrane and to should separate from the rest of foulants, what might be retained. Some authors have been studied the recovery of these detergents by NF processes using a spent single-detergent from a milk company (Fernández et al., 2010). In spite of that NF membrane (200 Da cut-off) maintains constant permeate flux rate (around 45 Lh⁻¹m⁻²) at 0.9 MPa, 70°C and 75% recovery rate after 1800 hours running, infrared studies demonstrated that some compounds present in the fresh single phase detergent are partially retained by the membrane.

8.2 Recovering of the other valuables constituents of wastewater of food industry

An overview of types and applications of membrane separation techniques to recover of proteins and functional compounds from wastewater cheese and fish processing are showed in this section.

Chollangi & Hossain (2007) evaluated the fractionation of dairy wastewater into lactose-enriched and protein-enriched streams using ultrafiltration membrane technique. Three membranes of MWCO of 3, 5 and 10 kDa of regenerated cellulose material were used to determine the efficiency of the process. The performance was determined under various processing conditions that include the operating temperature and TMP across the membrane and the concentration of lactose in the feed solution. It was found that the 3, 5 and 10 kDa membranes provided 70-80%, 90-95% and 100% recovery of lactose in permeate, respectively from made-up solution of pure lactose. The 10 kDa membrane results showed a 100% recovery of lactose from wastewater sample. Muro et al. (2010) worked with residual whey from a cheese industry, it was fractioned to recover proteins, lactose and
minerals by membranes process in filtration stages: UF and NF. The results of membrane process to treatment of whey depended on the operating conditions, but the temperature effect was greater in the ultrafiltration process. 80% of proteins from whey were recovered with the membrane of 15 kDa operating to 2.4 Lh\(^{-1}\) to 30ºC and 1.5 bar. The NF process showed that the transmembrane pressure affect lactose rejection, obtaining itself 70% of yield with the membrane of 0.150 kDa, using a flow of Lh\(^{-1}\) to 25 ºC and 1.8 bar.

Respect to wastewaters from fish processing, effluents contain a large amount of potentially valuable proteins. These proteins can be concentrated by means of ultrafiltration (UF) and recycled into the fish meal process, improving its quality and the economic benefits from the raw material, whereas the treated water can be discharged into the sea or reused in the plant. An extensive review of the application of pressure-driven membrane separation processes in the treatment of seafood processing effluents and recovery of proteins therein was presented by Alfonso & Bórquez, (2002b). Two effluents from a fish meal plant located in Talcahuano, Chile, were characterized. A mineral tubular membrane, Carbosep M2 (MWCO = 15 kDa) was used in the UF experiments. The operating conditions were optimized in total recirculation mode, and the subsequent concentration experiments were carried out at 4 bar pressure, 4 ms\(^{-1}\), crossflow velocity, ambient temperature and natural pH. The results show that UF reduces the organic load from the fish meal wastewaters and allows the recovery of valuable raw materials comprising proteins.

Dumay’s work focuses on the treatment of washing waters coming from surimi manufacturing using ultrafiltration technology at a laboratory scale. Four membrane materials (poly-ether sulfone, polyacrilonytrile, poly vinylidene fluoride and regenerated cellulose) and 5 MWCO (from 3 to 100 kDa) were studied at bench laboratory scale using the pilot Rayflow® 100, commercialized by Rhodia Orelis. The investigation deals with the ability for membranes to offer a high retention of biochemical compounds (proteins and lipids) (Dumay et al., 2008).

9. Conclusions

Wastewaters produced in the food industry depend upon the particular site activity. Animal processors and rendering plants will generate effluents with different characteristics to those from fruit/vegetable washers and edible oil refiners (suspended/colloidal and dissolved solids, organic pollution and oil and greases as well as microbial contamination). MF and UF systems can reduce suspended solids and microorganisms, whilst UF/RO combinations can also remove dissolved solids and provide a supply of process water and simultaneously reducing waste streams. UF systems can get more than 90% reduction in BOD and less than 5 mg.L\(^{-1}\) in residual solids and less than 50 mg.L\(^{-1}\) in grease and oil. NF systems are being used in a number of applications thank to the quick development in new membrane materials. In case of RO process, BOD removal rate of 90-99% is possible providing a low cost controlled source of bacteria-free water.

The favourable characteristics (modular) of membrane technologies allow to use different techniques as it has be seen all along this chapter. These hybrid processes can include traditional techniques as centrifugation, cartridge filtration, disinfection and different membrane techniques building a “cascade design” very used in many of the applications reviewed. The risk of membrane damage due to the contact with particles, salt conglomerates, chemicals or others substances must be minimized to prevent short
membrane life. Operation parameters must be carefully selected to obtain good results, especially not to overpass maximum temperature and transmembrane pressures recommended by membrane manufacturers. From the point of view of each particular process, to work at permeate flow rates below critical flux will assure longer runs. Membrane operating optimization is another aspect of paramount importance. It seems likely that the application of membrane systems in the food industry will continue growing rapidly. In particular, wastewater treatments will become more important in the next years because of the increasing cost of mains water and effluent sewer disposal. A membrane wastewater treatment system can be a major contribution to a food sector and its introduction may feature as part of the continuous improvement plans within an environmental management system.

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The global food industry has the largest number of demanding and knowledgeable consumers: the world population of seven billion inhabitants, since every person eats! This population requires food products that fulfill the high quality standards established by the food industry organizations. Food shortages threaten human health and are aggravated by the disastrous, extreme climatic events such as floods, droughts, fires, storms connected to climate change, global warming and greenhouse gas emissions that modify the environment and, consequently, the production of foods in the agriculture and husbandry sectors. This collection of articles is a timely contribution to issues relating to the food industry. They were selected for use as a primer, an investigation guide and documentation based on modern, scientific and technical references. This volume is therefore appropriate for use by university researchers and practicing food developers and producers. The control of food processing and production is not only discussed in scientific terms; engineering, economic and financial aspects are also considered for the advantage of food industry managers.

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