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### Membrane processing technology in food industry: Food processing, wastewater treatment and effects on physical, microbiological, organoleptic and nutritional properties of foods

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**Membrane processing technology in food industry: Food processing, wastewater treatment and effects on physical, microbiological, organoleptic and nutritional properties of foods.**

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## 1. ABSTRACT

Membrane processing technology is increasingly used nowadays in a wide range of applications (demineralization, desalination, stabilization, separation, deacidification, reduction of microbial load, purification, etc.) in food industries. The most frequently applied techniques are electrodialysis, reverse osmosis, nanofiltration, ultrafiltration and microfiltration. Several membrane characteristics such as pore size, flow properties and the applied hydraulic pressure mainly determine membranes' potential uses. In this review article the basic membrane techniques, their potential applications in a large number of fields and products towards the food industry, the main advantages and disadvantages of these methods, fouling phenomena as well as their effects on the organoleptic, qualitative and nutritional value of foods are synoptically described. Some representative examples of traditional and modern membrane applications both in tabular and figural form are also provided.

**Keywords:** membrane processing technology, food processing, electrodialysis, microfiltration, nanofiltration, reverse osmosis, ultrafiltration, demineralization, separation, microbial inhibition, nutritional value

## 2. INTRODUCTION

Membrane Processing Technology (MPT) is one of the most important fields of food science. As a result, food industries account for a significant proportion of the total production of membranes, of about 20 to 30% worldwide. Although these levels are quite high, there is an increase of approximately 7.5% annually in this field. All over the world, huge amounts of membranes are produced and find multi-purpose applications among which ultrafiltration (UF), nanofiltration (NF), reverse osmosis (RO) and microfiltration (MF) are the most extensively used. (Daufin et al., 2001). Electrodialysis (ED) can be described as an electro-membrane procedure in which ions are transferred through ion exchange membranes to different solutions under the effect of an electrical potential (Madzingaidzo et al., 2002). The main principle of UF process resides in the placement of a solution which contains large molecules, at one side of the membrane and the implementation of pressure, leading to removal of the solvent and retention of a highly concentrated solution. The enhanced concentration levels near the pores results in formation of a viscous layer, known as concentration boundary layer (Kumar & Upadhyay, 2000). RO is a membrane diffusion technique based on the implementation of pressure. Specifically, RO membranes retention level can reach up to 95–99% of the dissolved solutes (either organic or inorganic) filtered and the permeate water is characterized by excellent qualitative characteristics. As a result, RO is categorized as a concentration procedure (El-Salam, 2003). According to Gryta (2005), the Osmotic Membrane Distillation is a membrane distillation (MD) technique, applied at low temperature conditions. This process can be implemented with the aim to separate numerous aqueous solutions. The hydrophobic properties of these membranes

impede the penetration of aqueous solutions. As a result, only volatile compounds can pass through the membrane pores during this procedure.

The usage of hydraulic pressure, as the impelling cause of filtration, can be defined as a basic characteristic underlying the differentiation of the most often applied membranes such as MF, UF, NF and RO. However, the pore size and the properties of each membrane determine the size of particles separated according to their size or mass (Coutinho et al., 2009). Nowadays, MPT is applied in a wide range of food processing applications. It is important to mention that every type of membrane has its own advantages and disadvantages, which determine its potential applications. The purpose of membranes' application in food industries as well as the characteristics of various filtrated foods differ significantly. A synoptical presentation of these characteristics is given in Tables 1 to 5.

### 3. ELECTRODIALYSIS

According to Lonsdale (1982), electrodialysis has found several applications such as the separation of solutions from electrolytes (mainly applied in water desalination), selective retention or separation of non-electrolytes, oxidation effects minimization or formation of oxides and bases, double decomposition chemical reactions etc. The main principle of ED functioning is the separation of ions from an aqueous solution through the application of membranes and electricity. Anion and cation exchange membrane systems are stabilized between negative and positive charged electrodes. During the passage of a substance comprising ions from the channels of the system, the  $\text{Na}^+$  ions move toward the negatively charged electrode whereas the



Cl<sup>-</sup> ions move toward the positive. Since membranes are impermeable to ions, the latter are accumulated at both sides of the system, depending on their load, while the ionic charge is reduced at the remaining partitions (Pourcelly, 2000; Vera et al., 2009). Numerous uses of ED in food production are of considerable financial importance. A typical example is the demineralization of cheese whey products. Intensive investigations are conducted on the use of the method in procedures such as the deashing of molasses or the reduction of fruit juice acidity (Strathmann, 2000). The flux  $J_s$  parameter and a solution  $J_v$  across membranes at current density  $I$  can be described with the following equations:  $J_s = \lambda (I/S) - \mu (C'' - C')$  and  $J_v = \phi (I/S) + \rho (C'' - C')$ , where  $\lambda$  is the overall transport number,  $\mu$  is the overall solute permeability,  $\phi$  is the overall electro-osmotic permeability and finally  $\rho$  is the overall hydraulic permeability (Tanaka, 2009). RO is a membrane process becoming of increasing interest in the food industry, which presents several advantages with respect to evaporation and freezing, the other two techniques most widely used for this purpose. As the process is carried out at low temperatures, minimum thermal damage is caused. Other advantages are the lower energy consumption and the lower capital equipment costs (Alvarez et al., 1997).

### 3.1. Desalination

ED finds application in industries of biological products as well as in the wastewater treatment of different industrial plants. The procedure is used for water desalination with the aim of producing potable water, foods with reduced salinity and for the commitment of salt from various sources (production of table salt from seawater, wastewater desalination etc.) (Pourcelly,

2000). Much investment is directed to the application of ED for binding salt from brackish water (Davis et al., 2006).

During processing of fish and seafood, large quantities of effluents are produced. These by-products if discharged untreated, contribute significantly to environmental contamination. However, many aquatic solutions derived from thermal processing of such products, may contain aromas of high economic value. According to Cros et al. (2006), processing of these byproducts initially with ED (removal of salts) and afterwards with RO (retention of salt), is capable of collecting the initial aromas. Specifically, the electrodialyzer P1; EIVS, Le Vésinet, France, was used operating in a batch recirculation way at  $175 \text{ Lh}^{-1}$  at  $20 \pm 2^\circ\text{C}$ . After the procedure, membranes were cleaned ( $0.1 \text{ M HCl}$ , 20 min -  $3 \text{ g L}^{-1} \text{ NaOH}$  solution, 20 min) to remove foulants. RO processing was carried out with a tubular membrane made of polyether sulfone-polyamide membrane. Maximum product velocity was about  $2.5 \text{ ms}^{-1}$  (50 bar). It was reported that the method was of low cost and cleaning (circulation of a  $0.1 \text{ M HCl}$  solution for 20 min, application of deionized water and alkaline washing of  $3 \text{ g L}^{-1} \text{ NaOH}$  solution for 20 min) could be effectively applied to remove the film deposited on the membranes. Juice organic matter concentration was about  $5\text{-}20 \text{ g COD L}^{-1}$  while the mineral matter concentration varied within the range  $\text{g L}^{-1}$ . In the study of Cros et al. (2005), it was demonstrated that the collection of these aromas can be attained with the use of RO technology. However, due to the high osmotic pressure of these products, ED (constant voltage:  $20 \text{ V}$  and constant velocity:  $175 \text{ Lh}^{-1}$ , at  $20 \pm 2^\circ\text{C}$ ) is essential for the initial removal of salts (salt quantities can be limited from  $20 \text{ g L}^{-1}$  to  $2.8 \text{ g L}^{-1}$  for effectively preserving the valuable aromas). The ED method seems to be more applicable compared to diafiltration procedures, in the desalination of mussel cooking juices

because final products of higher quality can be produced. Specifically in this experiment, only a few minor changes in product characteristics in relation to the properties of the original raw material were observed.

In Japan the production of table salt by ED treatment of seawater in order to cover a large part of the country needs, is widespread. By processing, a salt solution of about 20wt% is produced, and then dried by thermal treatment. It is estimated that more than 1 billion kg of the product is collected on an annual basis through this method (Lonsdale, 1982).

### **3.2. Demineralization of dairy products**

ED can be implemented with positive results to demineralize a variety of dairy products. In the work of Andres et al. (1995) the demineralization potential of the skimmed milk by the application of ED (Stantech Inc., Hamburg, Germany and a semi-pilot scale stack from Eurodia, France) was analyzed. Two different types of membranes (PVC membranes and monovalent ion selective membranes) were applied for treatment of the product. The key feature of monovalent ion selective membranes is their ability to allow monovalent ions to pass while preventing the passage of larger molecular valence molecules. The experiments were carried out at 24°C. Almost the same percentage of demineralized products was observed for the two procedures (45% for the PVC membranes, 42% for monovalent ion selective membranes). Nevertheless, using the second method, the process was carried out in significantly lesser time without any losses of other ions. Furthermore, enhanced efficiency of 77% was reported using this method, while the same parameter was around 63% for PVC membranes. However, a major drawback

was the increased energy demands (27% increased) and fouling. The latter could be effectively prevented by applying low densities (20-25 mAcm<sup>-2</sup>) and cleaning the membranes. Both ED and ion exchange (IE) resins can be applied for the removal of salts from whey products. Greiter et al. (2002) examined the energy consumption of the two methods during their application in the recovery process of agents for the ion exchangers and wastewater process. The capacity of the system was evaluated at 45m<sup>3</sup> per day. The method involved the concentration and desalination of the processed product in triplicate. In the second method, the salts were removed at a rate of 99%. Implementation of ED resulted in significantly lower values in the parameters investigated. Specifically, the salts were removed at a rate of 90%. The method produced 1.25m<sup>3</sup> of waste water (8.1 kg ash and organic charge of 8.4 kg COD/m<sup>3</sup> of the product whereas IE resulted in production of 3.7m<sup>3</sup> of wastewater (36.3 kg ash and organic charge of 26 kg COD/m<sup>3</sup>). Furthermore, ED proved to be a less expensive method in terms of energy consumption (ED: 4.2 kWh for pumping, 5.38 kWh for the implementation of the required electricity, and 3.16 kWh for the reduction of the organic charge/m<sup>3</sup> whey, IE 0.15 kWh for pumping, 25.3 kWh for regenerants disposal, and 9.75 kWh for the reduction of the organic charge/m<sup>3</sup>).

### 3.3. Acids and bases production

Electrodialysis with bipolar membranes (EDBM) is one of the most widely used methods for the extraction of acids and bases from salt solvents. The method may require high energy consumption and can be effectively used to meet diverse needs (Recovery of HF and HNO<sub>3</sub>, NaOH from a stream containing Na<sub>2</sub>SO<sub>4</sub>, gluconic acid etc.) (Tongwen, 2002, Bazinet et al.,

1998). Provided that the anion-exchanger is towards the anode and the cation-exchanger is against cathode, the  $\text{OH}^-$  will be collected at the anode while the  $\text{H}^+$  will be collected at the cathode. Under these conditions, bipolar membrane systems retain a constant rate ( $\approx 10 \text{ kmol m}^{-3}$ ) of negatively or positively charged ions in the appropriate sides of the system thereby causing correspondingly the formation of acids and bases (Fidaleo & Moresi, 2006). ED procedure was used for the formation of citric acid from sodium citrate, with application of bipolar membranes made of polyphenylene oxide (PPO). The feasibility of the procedure was examined with a laboratory ED-cell (functional area of  $0.20 \text{ m}^2$ ). After detailed examination bipolar membranes' characteristics, the data on the accumulation of  $\text{Na}_2\text{SO}_4$  and sodium citrate were analyzed as concerns the transport and exchange of ions. It was estimated that, ideally, the concentration should vary between 0.5–1.0 M for the  $\text{Na}_2\text{SO}_4$  and 0.25–1.5 M for the sodium citrate. Maximum quantities of concentrated substance ( $30 \text{ L}^{-1}$ ) can be achieved at 0.5 M  $\text{Na}_3\text{Cit}$  - 0.5 M  $\text{Na}_2\text{SO}_4$  within less than 200min, at  $25^\circ\text{C}$  (Tongwen & Weihua, 2002).

### 3.4. Lactic acid separation

The production of lactic acid can be achieved by either chemical synthesis or producing it by fermentation. However, the formation of lactic acid by fermentation is a quite complicated and expensive process. A bench-top system equipped with 20 cell pairs (20 cation-exchange membranes - CR61 CZL-386 and 20 anion-exchange membranes AR103 QZL-386 - effective area:  $230 \text{ cm}^2$ ) was applied for the examination of ED process of several model solutions of this acid at numerous concentrations, changes in pH and different methodologies. As concerns the

batch recycle mode, the amount of acid flowed, showed a linear increase with time. The lactate flux was measured at  $328\text{--}456\text{ g m}^{-2}\text{ h}^{-1}$  and reduced with a parallel increase in feed concentration or pH. The water flux through ED was calculated at  $2.79\text{--}3.83\text{ l m}^{-2}\text{ h}^{-1}$ . The values of this parameter decreased with increase in feed concentration and did not remain stable during changes in pH. These levels of water transportation reduced the stream concentration down to  $135\text{ g liter}^{-1}$  (feed concentrations:  $45\text{--}95\text{ g liter}^{-1}$ ) (Yen & Cheryan, 1993). Repeated batch ED to recover lactic acid was applied in combination with solution of the same acid or fermentation broth. In all processes lactate fluxes reached up to  $7.0\text{ moles/m}^2\text{ h}$ , lactate was almost fully recovered ( $>99\%$ ) and the energy expenditure/ unit lactate during transport did not exceed  $0.25\text{ kWh/kg}$ . Application of ED in the aim of producing the fermented products, and addition of  $100\text{ gL}^{-1}$  glucose resulted in formation of less than  $92.4\text{ gL}^{-1}$  of the acid was formed (production rate:  $0.67\text{ gL}^{-1}\text{ h}^{-1}$ ). Furthermore, after application of ED in combination with  $150\text{ gL}^{-1}$  whole-corn flour hydrolyzate and  $5\text{ gL}^{-1}$  corn steep liquor, there was a significant enhancement in the productivity of the acid and the optimal cell growth by 2.5 and 1.8 times, respectively (Wee et al., 2005).

### 3.5. Tartrate stabilization of wines

Several experiments have shown that ED process can effectively contribute to stabilization of white, rose and red wine products without greatly affecting other sensorial properties of these products. It was clearly demonstrated that although cold treatment can also lead to stabilization of these wines, ED (total surface:  $25\text{ m}^2$ , treatment capacity:  $25\text{ hLh}^{-1}$ ) is associated with

deionisation frequencies of 26%, as concerns Fino wine and up to approximately 20% in wines such as Medium and Cream wines (Benítez et al., 2003). The function mode of ED is based on ion exchange and is completed in one step. This mode is inconsistent with the traditional ion exchange methods as they take place in two stages (including a stage of resin regeneration). In ED, the wine moves through rectangular channels the boundaries of which are defined by membranes. The function of these membranes is based on their permeability to ions and cations. The ions move through the membranes with an impetus from external electric field. The removal of ions results in a wine of reduced ionic charge (Gonçalves et al., 2003).

### 3.6. Vinasse demineralization

Moreover, ED using ion exchange technology has been implemented with positive results for the removal of a large portion (70-85%) of ash from vinasse characterized by a density of 5-15 mA cm<sup>-2</sup> at 35°C (vinasse of 6-2°Brix). After applying ED for vinasse treatment, a decrease in values of pH was observed. According to Fidaleo & Moresi (2006), in vinasse demineralized at a rate of 80% removal of all K<sup>+</sup>, significant decrease of Na<sup>+</sup> (52%), Ca<sup>2+</sup> (40%), and Mg<sup>2+</sup> rates (19%) were observed. In addition, demineralization of vinasse at a rate of 76% reduced the percentage of SO<sub>4</sub><sup>2-</sup> by 38%. According to Milewski & Lewicki (1988), demineralization of vinasse by about 80% resulted in complete removal of K<sup>+</sup> ions and drastic reduction of Na<sup>+</sup>, Ca<sup>2+</sup> and Mg<sup>2+</sup> at a rate of 52%, 40% and 19%, respectively. During the process there is no change in the concentration of betaine while the increase in the percentage of total amino acids is obvious in the final product. Potential increase in demineralization results in enhanced energy costs. To achieve demineralization rate of about 75%, approximately 10 Whg<sup>-1</sup> of ash were demanded.

### 3.7. Advantages and costs

ED method is distinguished over other methods by a number of advantages, the most important of which are the reduced environmental impact, the one-stage purification and concentration and technology simplicity. It is also distinguished by low energy consumption, easy implementation, immediate results and conduction at low temperature levels. Among the most obvious drawbacks, the high cost of membranes and their relative fragility can be referred (Elías-Serrano et al., 2007). The determination of the final cost of the process significantly depends on membranes' morphology. The morphology of the membranes has also a significant effect on their characteristics, the feed properties and the characteristics of the final-filtered product (Tongwen, 2002). In fact, the potential high procuring cost of a specific electromembrane (ca MLit  $0.6\text{m}^{-2}$ ) and the high-energy consumption (Lit 100kWh) may prevent the use of membranes for industrial production of high-added value products. However, the retention of harmful microbial products (normal market prices  $\approx$  Lit 2500-4000/kg) by ED can become the first choice of industries, provided that gypsum removal will remain a serious environmental issue. According to Moresi & Sappino (1998), the expenditure required for the retention of mono-sodium citrates in an industry that manipulates 2500 metric tons/year and uses  $100\text{gd}^{-1}\text{m}^{-3}$  of citric acid (at pH 3-7)  $c_{ee} = \text{Lit } 100\text{k}^{-1}\text{Wh}^{-1}$  and  $c_s = \text{Lit } 2000\text{kg}^{-1}$ , is estimated at Lit 1900  $\text{kg}^{-1}$ , the expenditure required for the retention of di-sodium citrates are estimated at Lit 774  $\text{kg}^{-1}$ , while for the retention of tri-sodium citrates the estimated cost amounts to Lit 565  $\text{kg}^{-1}$ . This fact indicates that the separation of citric acid from clarified fermentation broths with ED can be



lower if the acid is initially converted into tri-sodium salt.

#### 4. REVERSE OSMOSIS

A composite RO membrane is a film composed by two layers and produced in two consecutive stages. In the first production stage of such a membrane, a non-selective layer distinguished by a relatively increased thickness and large number of pores is formed. In the second stage, a second very thin layer is applied over the first. In contrast with “asymmetric” RO membranes (two chemically similar layers formed in one stage procedure), the chemical composition of the two layers differs significantly. An asymmetric RO membrane is composed by thick layers of the same chemical composition with another porous layer attached to them (Petersen, 1993). The diffusion of substances through RO membranes is achieved by pressure application. The filtration efficiency of various carbonate or non-carbonate solutes can reach up to 95-99% resulting in the production of water with exceptional qualitative characteristics. According to Sundaramoorthy et al. (2011), solvent flux  $J_v$  and solute flux  $J_s$  of RO membranes can be represented as  $J_v = A_w (\Delta\Pi - \Delta\Pi)$  and  $J_s = B_s (C_b - C_p)$ , where  $a_w$  factor represents the solvent transport coefficient,  $B_s$  represents the solute transport coefficient while  $\Delta P$  can be described as the pressure difference between the two sides of the membranes. The osmotic pressure difference  $\Delta\Pi$  between the two sides of the membrane can be described as  $\Delta\Pi = \gamma T (C_b - C_p)$  where  $\gamma$  is the gas constant,  $T$  represents the temperature value and  $C_b$  and  $C_p$  are the solute concentrations on each side of the membrane. The permeability over time of RO, UF and NF membranes during the filtration of grape must, depectinised bergamot juice and rude prolactine extract, respectively, is presented in figure 2.

#### 4.1. Water treatment

RO is widely applied for processing raw and wastewater as it is economical and can be carried out in room temperature (Tödtheide et al. (1997). Specifically, aromatic polyamide membranes (Toray: TR70-2514 F, membrane area:  $0.7\text{m}^2$ ) could effectively reject  $0.026\text{molL}^{-1}$  NaCl (99%) at 14.4 bar. At 18 to  $23^\circ\text{C}$  (10 bar) and feed flow of about  $2.5\text{Lmin}^{-1}$  it was shown that alterations in the retentates obtained were not due to enhancement of total concentrated solutions. RO is among the most innovative methods of water treatment and widely applied for treatment of wastewater, and desalination or reduction of salinity of brackish or seawater to meet the needs of fresh water for drinking or industrial operations. A wind-driven RO method was coined and used to effectively reduce the rate of nitrates from culture water of tilapia in Coconut Island. It was demonstrated that, through this process, freshwater can be treated and reused at a flux of 228–366 L/h pending on wind speed. The method proved highly effective (reducing undesirable substances from 90% to 97%) with a recovery rate of 40–56%. It was estimated that under real conditions the availability of  $1.0\text{ m}^3$  of water would have an average cost of 4 US\$ (Qin et al., 2005).

#### 4.2. Applications in dairy industry

Dairy industries require huge amounts of water for their operation and as a result, a large portion of wastewater causing environmental pollution comes from these industries. A  $540\text{ m}^2$  RO membrane system is required for processing  $100\text{ m}^3\text{d}^{-1}$  of wastewater and recovering the

highest percentage (95%). The RO procedure was applied with a flux of around  $11 \text{ Lh}^{-1} \text{ m}^{-2}$ . Total Organic Carbon (TOC) of treated water was calculated to be less than  $7 \text{ mgL}^{-1}$  and was mainly performed to lactose (76-100%). After purification, water was low mineralised (conductivity  $<50 \mu\text{Scm}^{-1}$  associated mainly to monovalent ions;  $\text{Na}^+$ ,  $\text{K}^+$  and  $\text{Cl}^-$ ). With the use of RO, water with qualitative characteristics close to vapour condensates (formed through drying procedures) can be produced, allowing its reuse for processes such as heating, cleaning and temperature reduce (Vourch et al., 2008). The application of RO in the dairy industry focuses mainly on the separation of waste solids from the water used for the operation of this industry. This procedure can be used to achieve two main objectives: firstly to reduce the biochemical oxygen demand (BOD) of byproducts in order to reduce the cost of water treatment and secondly to export of high added value solids which can be dehydrated and disposed for feeding requirements. Both objectives target to increase the revenue of the company. Moreover, since the final RO product derived from the treatment of several dairy products is distinguished by low BOD ( $30\text{--}300 \text{ mg l}^{-1}$ ), it can be most effectively applied for cleaning processes (El-Salam, 2003).

RO as well as other membrane techniques, the operation of which is based on pressure application, have been extensively applied for concentration and separation of whey protein load. Usage of RO requires the application of one sole procedure. This method can be used to pre-concentrate whey, as preparative step for effective spray drying, and for reduction of transport costs.

The benefits of concentration achievement using RO can be summarized as follows:

- Less expensive transportation and maintenance of whey,
- Preconcentration method during the production of whey-derived powder, demineralized whey

powder, and whey cheeses

-Improving the performance of UF membranes for the concentration of whey proteinic load (El-Salam, 2003).

### 4.3. Concentration of fruit juices

RO can be applied for preconcentration of fruit juices. The method can be used instead of high temperatures. In this way, the qualitative degradation of the product due to exposure to heat is significantly reduced and the process becomes of lower cost. In the work of Álvarez et al. (1998), the RO permeation of apple juice aromatic ingredients was examined. The RO membrane used is a spiral filter made of aromatic polyamide and with salt rejection coefficient of 99.2 %. The process was examined under pressure conditions of around 1.5–3.5 MPa and flow rates of 200–600  $\text{lh}^{-1}$ ; Reynolds numbers: 100 - 300, at 25°C. The quantity of rejected substance increased at higher flow conditions. The results were as expected for most of the hydrophilic substances, but fairly lower rejected material, in comparison with the predicted one, was observed for the least hydrophilic substances after the experiment.

Recently, a strong interest of researchers was shown on the applications of RO in juice concentration and aromas retention (Pozderović et al., 2007). Evaporation of fruit juices can result in significant losses of volatile compounds. However, the majority of these valuable components can be retained during concentration mediated membrane procedures. Use of RO (Lab Unit M20 - Dow Denmark Separation Systems De Danske Sukkerfabrikker, Copenhagen, Denmark) with the aim of concentrating apple juice esters and aldehydes was examined. It was found that this method can be successfully applied for the concentration of solutions of ester

(0.1% v/v of ester model solution (ethyl acetate, butyl acetate, ethyl butyrate, at 10, 20, 30, 40 bar) and aldehyde (0.1% v/v and 0.05% v/v, at 40 bar) characterized by various forms and properties. During the application of HR 98 PP (permeability %NaCl: <2.5, water flux: 105-145 Lm<sup>-2</sup>h<sup>-1</sup>, pH: 2-11, temperature: 0-60°C, pressure: 0-60 bar) membrane, higher commitment degree of acetate, butyl acetate, ethyl butyrate and ethyl 2-methyl butyrate was observed than with the use of HR 95 PP (permeability %NaCl: <5, water flux: 115-155 Lm<sup>-2</sup>h<sup>-1</sup>, pH: 2-11, temperature: 0-60°C, pressure: 0-60 bar) membrane. On the other hand, using the latter, better results in the commitment of hexyl acetate, amyl acetate, iso-amyl acetate, iso-amyl propionate and hexanal and trans-2-hexenal were observed. The method had better results when aldehyde quantities and molecular mass were at higher levels (concentration of hexanal of about 0.01%v/v was accompanied with organic compounds passage of about 77.25% while at concentration of 0.05%v/v, 63.74% of organics permeate was observed). The highest retention rates were observed when concentrated monounsaturated (double bond) aldehyde was applied. Application of higher pressures increased the efficiency and effectiveness of the membranes (Pozderović et al., 2006).

Many studies examined the applicability of RO to concentrate tomato juice or serum (Yildiz et al., 1993). Tomato juice can be distinguished by a wide range of characteristics depending on the source region of the fruit, growing environment and the applied treatment. Nevertheless, processing of the fruit with RO displays similar effects regardless of its origin source. Initially, concentrated tomato juice was produced with centrifuging or ultra filtrating the food, with the aim to removing the dietary fibers and other undesirable materials and then completing the process with RO. The resulting product is mixed with the non-soluble

components. The above procedure has not been used in bulk by the industries (El-Salam, 2003). RO application to food industries aims at significant savings. Concentration to 20.69 and 20.36% soluble solids of Napoli VF tomato juice was effected with RO (tubular membranes: 1.4 cm inner diameter, 4.5 m long, output of up to 50 l m<sup>-2</sup>h<sup>-1</sup>, pressure: 60 bars at 35°C) and evaporation, correspondingly. The most important factors considered were titratable acidity, soluble solids, pH, invert sugar, ascorbic acid, pectins, calcium, iron and copper. The measurements carried out included Hunter L\*, a\*, b\* values, Bostwick consistency, black specks and sediment determination. Besides the measurement of total microbial load (incubation at 35°C for 48±2h), special attention was given to the evaluation of Howard mould (12 cfu ml<sup>-1</sup> with RO and 32 cfu ml<sup>-1</sup> with evaporation), *Bacillus coagulans* (absence after treatment) and coliform microbes (absence after treatment). Organoleptic examinations were related to colour evaluation, structure consistency and flavor characteristics. An important (p < 0.01) alteration among qualitative properties of the concentrated products was recorded. Except for the better microbiological and sensorial quality of RO products, a sample of fifty individuals also showed apparent preference in colour (RO: L=27.9 - a=30.8 - b=14.8, evaporation: L=23.6 - a=37.0 - b=13.0) and flavor characteristics of tomato products treated with RO. 46mg/100g of ascorbic acid was detected in RP-processed juice while only 37 mg/100g of the same vitamin was detected in evaporated tomato juice. However, pectin, Ca, Fe and Cu of RO-processed juice amounted to 0.18%, 149ppm, 25mgkg<sup>-1</sup> and 27.5 mgkg<sup>-1</sup>, respectively, while in evaporated product, the same substances were included in the following quantities; pectins: 0.26%, Ca: 136ppm, Fe: 30mg/kg and Cu: 35mgkg<sup>-1</sup> (Yildiz et al., 1993).

The blackcurrant is a popular fruit containing a number of ingredients greatly acclaimed

for their biological activity. Szép et al. (2009) prepared a concentrated blackcurrant juice with RO to preserve the valuable components of berries. A Paterson Candy International (PCI) apparatus was used for juice concentration, equipped with a tubular B1 RO membrane module. The active membrane area was 0,9 m<sup>2</sup>. The juice was concentrated at 20°C and 60 bar pressure. The permeate flux was treated with two pectinolytic enzyme preparations to investigate the implementation of the RO for blackcurrant juice concentration. For the control sample, The lowest permeate flux was reported for the control sample whereas the maximum TSS of the concentrate reached was at 22.5° Brix. Pretreatment of concentrated juice with Cellobiase from *Aspergillus niger* resulted in the highest permeate flux.

#### 4.4. Dealcoholization

Many methods have been extensively tested to reduce the proportion of alcohol in various beverage industries. Towards this end, RO membrane systems have been successfully applied either industrially or on a large scale in many countries (El-Salam, 2003). Generally speaking, this procedure is quite demanding in terms of equipment required and increases the cost. Thus, it is inexpedient for the treatment of products with a low alcohol percentage (<0.45%). The original extract and carbohydrates are of low concentration and the beverage has a rather appealing taste. Specifically, products of this type with higher pH levels result in concentration polarization and as a result fouling is enhanced by preventing the normal operation and profitability of the membrane. Nevertheless, RO method can be significantly improved under the condition those compensative abilities are advanced (Pilipovik & Riverol, 2005).

#### 4.5. Advantages, disadvantages and costs

The main benefits arising from the use of RO concentrated products are the advanced qualitative characteristics (better preservation of nutrients and sensorial characteristics) due to treatment in low temperature conditions, the low cost operation and the relative ease of proceeding. According to Jesus et al. (2007), the concentration of orange juice by RO (filtration area:  $0.72 \text{ m}^2$ ) was investigated at 20, 40 and 60 bars. The soluble solids were about 16 (20 bar), 28 (40 bar) and  $36^\circ\text{Brix}$  (60 bar). The ascorbic acid quantities augmented from 29.3 mg ascorbic acid/100 g (single strength juice) to 53.9, 82.7 and 101.1 mg/100 g, in the permeates received at 20, 40 and 60 bar, respectively (permeate flux was  $28 \text{ Lh}^{-1} \text{ m}^{-2}$  at 60 bar,  $20 \text{ Lh}^{-1} \text{ m}^{-2}$  at 40 bar and  $11 \text{ Lh}^{-1} \text{ m}^{-2}$  at 20 bar). It was shown that filtrated product preserved effectively its original organoleptic characteristics.

However, RO has also some drawbacks. To start with, the preparation before processing is necessary to be carefully controlled and the application of different chemicals may raise significantly the cost of the process. Moreover, there is no production possibility of sterile water and membranes have a limited lifespan (3-5 years) and, as a result, represent a significant proportion of the costs of the procedure. Furthermore, water low in solids can cause problems in piping and equipment and, finally, application of high pressure is required in the initial stage of the procedure (Bena, 2004). The most important drawback is the low efficiency of concentrated product achieved with RO compared with thermal evaporation. This is due to the conditions of increased osmotic pressure (characteristic of fruit juices) that reduce the efficiency of the process (Jesus et al., 2007).



## 5. NANOFILTRATION

In recent years, NF has been extensively used in industrial scale for separating particles that bear or not electric charge in water solutions (Wang et al., 2009). When silage juice was nanofiltered (PES10, N30F and MPF36) with the aim of purifying lactic and amino acids, the mean flux was about  $6.5 \text{ L m}^{-2} \text{ h}^{-1}$  while discoloration reached up to 80-99%. The cross flow velocity reached at  $2.7 \text{ ms}^{-1}$  and average flux of the optimum NF method (PES10,  $6.6 \text{ L m}^{-2} \text{ h}^{-1}$ , organic, at 3.5 MPa) fluctuated at the same levels as the flux of the organic UF membrane (PES004H,  $6.5 \text{ L m}^{-2} \text{ h}^{-1}$ , organic at 0.7 MPa). Fouling was found to be the main disadvantage of the method resulting in high costs since effective cleaning could be achieved at  $50^\circ\text{C}$ . It was shown that PES10 purified products of high quality without high losses of amino acids at a flux of  $6.6 \text{ L m}^{-2} \text{ h}^{-1}$  (Koschuh et al., 2005). NF is often applied in the following separation processes; desalting of brackish and seawater, retention of minerals from wastewater, effluents processing of industries correlated to manufacturing of clothing and removal of contaminants from companies producing medicines, edible and organic products. Prescribing these separation processes is extremely important for these industries. As a result, several models have been tested for parallel determination of steric results and control of membranes' permeability (Bowen & Welfoot, 2002).

The most important advantages of the NF membranes are the reduced cost in comparison with RO membrane systems, as well as increased retention capacity of organic matter when compared with UF (Bessarabov et al., 2002). Fouling is the most severe problem of NF due to the several interactions that occur at nanoscale level thereby making the improvement of the

method very difficult. The disadvantages of fouling involve the pretreatment and frequent membrane cleaning demand, reduced recovery quantities and limited membrane lifetimes. It is therefore obvious that fouling is highly connected to several drawbacks such as concentrate treatment and unstable membranes (Van der Bruggen et al., 2008).

NF membranes made of polymers can be effectively represented by a solution-diffusion pattern. In this way, the flux of organic substances through this kind of membranes is described by the following equation:  $J \propto (V_m/\eta)(1/\phi^\pi \gamma_m)$  or  $J \propto (V_m/\eta \Delta\gamma)$ , where  $\Delta\gamma$  represents the difference in surface tension (mN/m),  $\eta$  represents the dynamic viscosity (Pas),  $V_m$  is the solvent molecular volume ( $\text{m}^3/\text{mol}$ ) and  $\phi$  is used to quantify the membrane-solvent interactions (Van der Bruggen et al., 2008).

### 5.1. Water treatment

MPT has been extensively applied for the purification and processing of food industry effluents. Wastewater can cause rapid concentration of the membrane layer and increase fouling rates. This phenomenon can be amplified when the product enters the membrane system under high pressure. In whey production, treated whey is hardly achieved due to its increased organic concentration ( $100,000 \text{ mg O}_2\text{L}^{-1} \text{ COD}$ ). A single stage NF system presented the optimum characteristics producing disposing products with COD of about  $2,787 \text{ mg O}_2\text{L}^{-1}$  while rejected proteins reached up to 88% (permeate flux  $30.8 \text{ L/m}^2\text{h}$ ) (Yorgun et al., 2008). Due to the ever-increasing processing costs of water and wastewater, the small food and beverage industries invest in developing low-cost methods to produce water of potable quality from the effluents

water of their production line. One way of achieving this goal is the use of a bioreactor, in conjunction with membranes, followed by treatment with a NF/US method capable of fully disinfecting the wastewater. The procedure has been experimentally tested ( $\leq 1.5 \text{ m}^3\text{h}^{-1}$ ) in a fruit juice industry. As concerns the bioreactor, COD removal percentage was estimated to be higher than 95%. Upon completion of the whole process, the qualitative characteristics of the water were equivalent to drinking (in accordance with the requirements of the German Drinking Water Act - DWA). After granting license, the water could be reused to meet the needs of food production (Blöcher et al., 2002).

Some of the most important uses of NF do occur in the area of water softening and salts fractioning from low molecular mass compounds. The potential uses of the technology are constantly increasing. Thus, NF can now be used complementary to salt treatment, in the retention of sulfate during the filtration of seawater as well as to processing of petroleum products (Vellenga & Trägårdh, 1998). According to Song et al. (2004), “the  $\text{H}_2\text{O}_2$ /UV oxidation of source water prior to NF showed potential for the following: (i) mitigation of flux decline due to membrane fouling, (ii) removal of the pesticide alachlor and hydrogen sulfide, and (iii) improvement in membrane cleanability”. According to Wang et al. (2005), natural organic matter (NOM) can act as a determining factor in the causation of fouling during water purification. Although application of NF in wastewater processing and reuse can be considered significant, several problems appear to hinder its widespread use. The most important of these, is the accumulation of foulants.

## 5.2. Separation processes

NF procedure can be effectively used to separate different substances from liquid streams of organic nature. Through this process both cost and environmental pollution can be minimised. One of the most important applications of NF is in solvent exchange (Veríssimo et al., 2007). Furthermore, NF can be applied for the removal of undesirable or hazardous substances of low molecular weight. The technology was used on an industrial unit to bind DDT [(1,1-bis(4-chlorophenyl)-2,2,2-trichloroethane)]. Several parameters, including the amount of the substance in the initial sample, pH and percolation rate, were examined in order to determine their effect on method effectiveness. It was proved that DDT was effectively captured. Increasing pressure resulted in an increase in saturated rate. Concentration of about  $77 \mu\text{gL}^{-1}$  led to depletion of the substance within half an hour while concentration of about  $5 - 20 \mu\text{gL}^{-1}$  resulted in binding a lower percentage of the substance (from 95 to 85%). During the process, the feed flow was maintained at  $35 \text{ Lh}^{-1}$  (0.24 MPa). When the DDT concentration at the beginning of the process was about  $5 - 20 \mu\text{g/L}$ , the rejected material reached up to 95 to 85%. Rejected quantities were reduced by limiting the initial DDT concentrations. Provided the maintenance of a steady recovery rate, increased flux may lead to lower quantities of rejected substance (Pang et al., 2010). Silage juice consists of various compounds and the retention of useful substances, such as lactic acid and several proteins, is very important. In the study of Koschuh et al. (2005), NF was applied for the purification of lactic acid and amino acids of high value. 3 different NF membrane systems (PES10, N30F and MPF36) made of organic materials were applied and evaluated in order to find the most appropriate method. The peak flow observed during the treatment was around  $6.5 \text{ Lm}^{-2} \text{ h}^{-1}$ . It was demonstrated that the decrease of color intensity

reached at 80–99% and the optimum results were demonstrated after the implementation of PES10, which was proposed for use in substances' retention from silage juice. Applications of NF for the separation of different valence ions, organic compounds from aqueous solutions etc. could experience wide acceptance by food industry. Nevertheless, the method should be significantly improved to increase efficiency and solve any problems encountered (Van der Bruggen et al., 2008).

### 5.3. Applications in dairy industry

Frappart et al. (2006) examined the retention of lactose and several milk proteins using UF. Carbon oxygen demand (COD) and limitation of ions' rate in waters received from the production process, simulated by UHT skim milk (dilution 1:2) with initial levels of COD of about 36,000 mgO<sub>2</sub> L<sup>-1</sup> were also evaluated. To carry out the process a flat circular metal structure (radius: 7.25 cm), either smooth or provided with vanes, was used. The structure rotated near a Desal 5 DK membrane. The filtered product was subjected to transmembrane pressure (TMP) of about 4000 kPa at 45°C (the values were among the following limits: from 130 L h<sup>-1</sup> m<sup>-2</sup> for a smooth disk at 1000 rpm to 230 L h<sup>-1</sup> m<sup>-2</sup> applying a disk with vanes at 2000 rpm). COD permeation presented the lowest values for the case in which the highest shear percentages and limitation with enhancing TMP (from 60 mgO<sub>2</sub> L<sup>-1</sup> at 1400 kPa to 22 at 4000 kPa) were shown. After the conduction of concentration tests (TMP of 4000 kPa, at 2000 rpm with vanes) a decrease in flux was caused after increase of volume reduction ratio (VRR) (= 100 L h<sup>-1</sup> m<sup>-2</sup> at VRR = 7.5). It was proved that by using the circular metal structure (6 mm vanes rotating at

2000 rpm), the highest VRR value was measured at 14.3, which indicates the existence of about 38% of dry substances.

#### 5.4. Applications in grape processing and oenology

Grape pomace is widely known for its high concentration of antioxidants. The commitment of these substances has been attempted using several technologies such as direct use, retention using traditional solvents, retention with supercritical CO<sub>2</sub>, or combination of methods. In the production of alcoholic beverages, grape pomace can be processed by distillation, procedure that leads to spirits production. This process leads to the formation of waste solids known as “distilled grape pomace (DGP)”. The product formed through application of pressure on grape pomace can be broken down into its components and evaluated for its content in antioxidants. Finally, the product can be processed with the aim of improving its quality and selling or using it for manufacturing other products. The methods used for this purpose include adsorption–desorption or application of UF and NF, succeeded by solvent retention of concentrates and adsorption–desorption of permeates. Nanomax 95, Nanomax 50 and Inside Céram membranes were applied to recover antioxidant substances from grape pomace processing effluents at 20°C. The permeates showed ABTS radical scavenging capacities of about 1.5–9.9 g of Trolox g<sup>-1</sup> of desorbed substance, and DPPH radical scavenging capacities that can be compared to BHA, with losses of about 1.94– 2.97 mM of vitamin C (Díaz-Reinoso et al., 2010). NF is one of the most effective methods and has been widely applied to increase the sugar levels in grape must (intended for manufacturing of wine). This method is highly accurate and does not cause

significant deterioration in the product quality (García-Martín et al., 2009).

The investigation into the potential applications of NF greatly concerns the wine industry. The various processes that occur during application of the method are directly dependent on membrane type and filtered product. Because of the complexity and the large number of materials that can be used, evaluation of filtering effectiveness is quite complex and data obtained in each procedure can vary significantly (Massot et al., 2008). According to Díaz-Reinoso et al. (2009), several important components of wine can be effectively concentrated using NF. These membranes can be used for permeation of  $H_2O$  or  $C_2H_5OH$  with better performance in comparison with other methods such as MF and UF. Although RO appears to have similar results with NF, it is more costly due to the increased pressure applied to membrane systems in order to carry out the process. Moreover, NF is not necessary to be applied in high temperature conditions, thus preserving the original qualitative characteristics of the wine. Nevertheless MF and UF are also applied by industries mainly to eliminate hazards (microorganisms etc.) that can potentially affect product safety (Banvolgyi et al., 2006).

### 5.5. Sugar processing

In the sugar industry, regeneration of waste disposal is difficult due to the increased amounts of sodium chloride and colored organics contained in the regeneration effluent. In the work of Cartier et al. (1997), spiral-wound NF membrane systems were applied in order to recover and recycle these high-salted solutions after their application in decolorization of sugar spirits. The method proved to be very effective since up to 74% of NaCl was removed whereas in

the case of water the corresponding maximum value reached 89%. Besides efficiency, the method proved to be less expensive than conventional methods. Nowadays, membrane applications in sugar processing and manufacturing have attracted the interest of researchers. In manufacturing of products such as sugar beets, one of the key issues is the retention of substances that do not contain sucrose. NF process (membrane made of polymers with MWCO of 0.5 kDa) was used to separate the colored solution of these substances from green sugary solution (39.2% d.m.). The effectiveness of the method at 30 and 50°C (5–30 bars) was evaluated. It was demonstrated that the procedure had better results at pressures of about 30 bars and flow rate around 300 to 400 l h<sup>-1</sup>. Under these circumstances, the color intensity was limited by about 76% (Gyura et al., 2005).

### 5.6. More applications of nanofiltration

More NF uses of economic importance are found in the field of soy juice processing, in fisheries and tomato products production as well as in the management of brewery liquid effluents. In the study of Luo et al. (2009), several NF systems (NF270, NF-, NF90, Desal-5 DL) were used for the retention of NaCl and concentration of valuable substances such as amino acids and aromas. The best results were reported with the use of NF270. It was found that the method was effective enough in terms of amino nitrogen and NaCl rejection, water consumption and operating pressure. It was demonstrated that the data extracted from mass balance equations were similar to those of the experiment. Liquid waste accumulated during the operation of seafood industry, an industrial field that requires huge amounts of water for its functioning, is



characterized by high proteinic load. The treatment of this wastewater is essential not only to protect the environment, but also for the retention and recovery of valuable substances contained therein. UF and NF can be applied toward this purpose (Alfonso & Bórquez, 2002).

According to Iaquina et al. (2009), during processing of tomato products (canning, concentration, production of pulp), large amounts of liquid wastewater (pH: 6.62, COD: 1200mgL<sup>-1</sup>, TOC: 340mgL<sup>-1</sup>) are formed. Cleaning and safe disposal of such wastewater can be done in a two-step process involving biological purification and application of NF (Desal-5 membrane, model DK2540, Osmotics) at 20±1°C accompanied with frequent flux measurements and taking into account the critical flux. It was clearly demonstrated that purified potable water can be produced by applying a recovery rate of about 90%. At 4.5 bars, a permeate flow rate of about 20.5 Lh<sup>-1</sup> was observed (initial flux: 13.4 Lh<sup>-1</sup>m<sup>-2</sup>). Accumulation of foulants can be prevented at permeate fluxes of ≤ 8.2 l h<sup>-1</sup> m<sup>2</sup>. Vast quantities of groundwater are required by the beer industry to meet various needs. Braeken et al. (2004) examined the effectiveness of NF application for the processing of brewery liquid effluents. The process aims at cleaning and reusing this water. Water coming from 4 industrial procedures (effluents processed with organic methods, glass container rinsing water, water used for cleaning the product manufacturing area and rinsing water of the bright beer reservoir) was processed by 4 kinds of NF membranes (UTC-20, UTC-60, Desal-HL-51 and Desal-5-DK). It was found that water processed by organic methods displayed the best results. In contrast, removal of organic substances was not efficient as concerns the maintenance of the required qualitative characteristics. During the experimental procedure (3h), a flux of about 10-40% was observed in 3 of the 4 membranes, which was considered insufficient. Desal-5-DK was the only one that presented enhanced flow in several

conditions of increased pH.

## 6. ULTRAFILTRATION

UF is a membrane process the driving force of which is pressure. In this method, membranes with suitable properties are used for the separation of large molecules and colloidal substances from liquids. The size of the molecules that can pass through the membranes depends on the diameter of membrane pores (De Bruijn et al., 2005). Substances with a molecular weight lower than 300 are easily dissolved in liquid solutions and characterized by increased osmotic pressure. During the application of UF, these substances penetrate the membrane as filtration depends on the size of the pores and not on osmotic processes. As a result, UF can be applied at low-pressure levels (usually 1-5 atm) (Lonsdale, 1982). Over the last 30 years, UF has been effectively used in a wide range of applications for the manufacture of various foods such as dairy products, products of plant origin, alcoholic beverages, sweets etc. This technology finds important application in treatment of water and effluents as well (Susanto et al., 2009).

According to Morão et al. (2001), “UF has the advantage of not requiring filter aid and flocculants as the conventional filtration process does. The filtrate quality is also another advantage of the membrane process because the permeates are completely free of suspended solids”.

UF is widely used in food manufacturing. However, the main disadvantage that characterizes the method and reduces, to some extent, its implementation in large industries, is blocking of pores. The methods of fouling treatment are not sufficiently developed because the

knowledge of the physicochemical properties of these membranes is not fully clarified yet (Rabiller-Baudry et al., 2006). The UF flux ( $J$ ) can be correlated with the proteins that are usually accumulated among the pores of the membranes (fouling) through the following equation:  $J = K \ln(C_g/C_b)$ , where  $K$  is the mass-transfer coefficient,  $C_b$  is the bulk concentration and  $C_g$  is a factor experimentally measured (Élysée-Collen & Lencki, 1997).

### 6.1. Applications in dairy industry

Milk is a mixture of substances that contains different concentrations of casein, a variety of proteins, fats,  $C_{12}H_{22}O_{11}$ , and various ionically charged salts. Although a large amount of milk is consumed pasteurized, an equally large amount is also used to produce milk powder or cheese. These dairy products are made through concentration of milk proteins. UF is widely applied to perform the above procedure (Rice et al., 2009a). According to Atrra et al. (2005), UF of skimmed milk carried out in membranes made of polyethersulfone (PES) (molecular weight cut-off: 5–10 kg molL<sup>-1</sup>) can be used for the evaluation of protein load prior to cheese production. The product passes through the pores of the membrane for 6–8 h at 50°C and then a water-rinsing procedure takes place. This second stage enables the enhancement of concentration of nutrients. Finally, the facilities are thoroughly disinfected both at low and high pH with alkaline chlorine solutions (Rabiller-Baudry et al., 2006).

Whey proteinic load retained by whey UF can be used to address a large number of needs. The type of proteins and the characteristics of the products determine their further use. The by-products of the process (separated via UF permeation processes) can be used in many food

industries. Atrá et al. (2005) provide information for methods of  $C_{12}H_{22}O_{11}$  concentration by application of NF or RO. These methods can be used in the manufacture of confectionery or pharmaceutical products. Specifically, the concentration of the light milk, concentration and processing of the whey proteinic load by UF, as well as the concentration and processing of ultrafiltered products by NF are able to effectively take place by using the membranes: FS10, SP015 and RA55. After the application of UF, rejected proteinic load did not exceed 92– 98% and permeation flux was about  $30 \text{ l m}^{-2} \text{ h}^{-1}$ ) (pressure levels of approximately 3 bars). UF of milk and whey proteins is effectively carried out at temperatures of about  $50^{\circ}\text{C}$  because under these conditions the viscosity levels of filtered product are relatively low. Application of increased temperatures can rapidly denature proteins and affect the effectiveness of the membranes.

## 6.2. Applications in fruit juice industries

During the last decades, UF is used in fruit juice manufacturing industries. The purpose of the membranes is focused mainly to clarify these products. According to Gökmen & Çetinkaya (2007), UF of apple juice is a quite economic and practical method of clarification. Research on the improvement of UF membranes resulted in substantial amendments like the construction of more resistant membranes capable of operating for longer periods of time, increased separation efficiency and resistance to chemicals. These characteristics make the method more and more attractive for use in fruit juice industries. On the other hand, products of this type, treated with UF, can be unstable, presenting alterations in the original characteristics of the product. The deteriorations observed include haze production and color changes. These phenomena are

attributed to the effect of polyphenols that, due to their size, penetrate the pores of the membranes and get accumulated in the final product, deteriorating its quality (Borneman et al., 1997).

### 6.2.1. Apple juice

The efficient apple juice clarification with UF was examined by De Bruijn et al. (2003). To carry out the procedure, a Carbosep® membrane (15 and 50 kDa) with tangential speed of 2 and 7 ms<sup>-1</sup> and driving pressure of about 150 and 400 kPa, was used. Accumulation of foulants, method effectiveness and qualitative characteristics of the final product were examined. The product flow through the pores ranged from 56 to 157 Lm<sup>-2</sup>h<sup>-1</sup>. According to the exported data, more than one combination of variables can optimize the efficiency of the method. Application of UF resulted in color enhancement and better clarity and turbidity. Degradation of the qualitative characteristics of the product was due to clarity decrease, turbidity and precipitation of solids at the bottom of the container after bottling. The clarity degradation factor was evaluated through mathematical models (zero-order reaction kinetics) and safe storage time was specified. After pasteurization (for 1/2h at 65°C) and application of UF, the product was effectively preserved for 5 months at 16°C (in the absence of light). Gökmen & Çetıkaya (2007) evaluated to what extent treatment with gelatin and bentonite, pressure (DP) and MWCO, before filtration, may affect the reduction or increase in flux during application of UF. The data related with the filtered volume were evaluated using De La Garza and Boulton's exponential model. The purpose of this study was the determination of the coefficient of foulants concentration (k)

and the filter resistance factor ( $R_m$ ). Enhancement of the rate of gelatin and bentonite applied for pre-processing of apple juice, resulted in the fluctuation of  $k$  at lower levels throughout UF procedure. It was concluded that under these conditions, a smaller percentage of foulants was accumulated on membrane pores allowing normal membrane function for longer periods of time. Variation of DP at higher levels resulted in an increase of  $k$  and  $R_m$ . In contrast, enhancement of MWCO reduced the effectiveness of both membrane purification and  $R_m$ . Nevertheless, MWCO of about 100 kDa brought the highest values of  $k$  thereby proving that the membrane is characterized by a strong trend for fouling.

#### 6.2.2. Sweet oranges

According to Cassano et al. (2009), modified poly(ether ether ketone) (PEEKWC) and polysulphone (PSU) hollow fibre (HF) UF membranes were manufactured using dry-wet spinning method (phase inversion procedure) and evaluated in terms of water flow rate through the pores, retention of dextrans and Scanning Electron Microscopy (SEM). The flux permeability was evaluated to determine whether it is appropriate for being used for clarification of Clementine mandarin juice product. The largest percentage of dissolved solids and the 2-hydroxypropane-1,2,3-tricarboxylic acid were retained. This process resulted in food production with improved appearance and color. PEEKWC systems presented a limited retention as concerns antioxidants (retention rate of about 21.8%) compared with the PSU membranes (retention rate of about 32.0%). Cassano et al. (2007b) investigated a cross-flow UF system equipped with polyvinylidene fluoride (PVDF) membranes for the clarification of blood orange

juice. During the experiment, the decreased or increased pressure applied to the membranes, the flow rate and temperature were evaluated. Examination of the obtained results revealed that at regular, fixed pressure and temperature conditions, the accumulation of foulants increases gradually depending on the applied flow rate. Investigation of the quality characteristics of products processed with UF revealed that orange juice after clarification was characterized by high quality similar to fresh product. The only disadvantage was the concentration of solids that were not dissolved. Total antioxidant activity (TAA) of the final product varied at slightly lower levels (2.5% lower) in comparison with the initial raw material.

### 6.2.3. Kiwifruit juice

In a study by Cassano et al. (2007a), raw kiwifruit juice treated with pectolytic enzymes was filtered using UF for clarification of the product. The experiment was carried out to assess the effects of applied pressure, axial flow-measure and temperature on flux rate of fluid through the membrane pores. It was demonstrated that flux fluctuated at higher levels when temperature ranged between 20-30°C and feed flow ranged from 300 up to 700  $\text{lh}^{-1}$ . The flux-pressure curves demonstrated no increase in permeate fluxes when TMP was higher than 90 kPa ( $\text{TMP}_{\text{lim}}$ ). UF application was used to effectively clarify the product by limiting the suspended solids and the turbidity of the product. After processing, the product contained slightly lower rates of vitamin C (84% of the original amount). Nevertheless, the antioxidant properties demonstrated losses of about 8%. According to the same author “cake layer and irreversible fouling resistances gave a minimum contribution to the total resistance (2.23% and 2.75%, respectively) while the

contribution of the reversible fouling was much more significant (29.4%)". The filtration capability of membranes was dramatically improved to 96% after chemical cleaning of the membranes. It was concluded that the main problem arisen by the operation of these membranes is the accumulation of foulants on their surfaces. Modified poly (ether ether ketone) (PEEKWC) membranes, made of hollow fibres (HF), were manufactured using dry–wet spinning method after a phase inversion procedure took place. The system was examined with SEM, H<sub>2</sub>O permeability measurements and in terms of dextran retention (R%). The effectiveness of HF membranes was experimentally tested for their ability to clarify kiwifruit juice. Under appropriate conditions, the preliminary flux permeation of 50Lm<sup>-2</sup>h dropped to 25Lm<sup>-2</sup>h<sup>-1</sup>. These data indicate the achievement of higher flux permeation in comparison with data mentioned in previous publications related to kiwifruit juice UF. The clarity of the final product amounted to 99% in the absence of suspended solids and the qualitative properties remained at high levels (Tasselli et al., 2007).

#### 6.2.4. Sugarcane juice

UF was recently used to clarify and purify sugarcane juice products. Ultrafiltered sugarcane juice has several advantages including a higher degree of clarity and lower viscosity and coloration. These properties greatly enhance the qualitative value of the product. Although, in recent years, the method has been applied in some industries, its use has not been widespread yet because of significant problems encountered during the operation process (concentration polarization and concentration of foulants on membranes). To overcome these disadvantages,



various efforts have been focused mainly on feed pretreatment, improvement of methods and use of various membrane materials. Moreover, different juice substances able to affect the membrane properties have also been investigated. The examination of the effect of sugarcane juice polysaccharides on the fouling of polymeric polysulphone and polyethersulphone membranes revealed that fouling led to a shift in the molecular weight cut-off of the membranes (Saha et al., 2006).

### **6.3. Poultry wastewater treatment**

Lo et al. (2005) evaluated the potential use of UF for the retention of proteinic load from poultry treatment effluents. Application of polysulfone membranes (30,000 molecular-weight-cut-off) isolated most of crude proteins limiting, at the same time, the COD of effluents at levels lower than  $200 \text{ mgL}^{-1}$ . The maximum flux levels during the use of membranes reached up to  $100 \text{ Lm}^{-2}\text{h}^{-1}$ . Evaluation of major factors such as pH (6.74), volumetric flow speed ( $683 \text{ mLmin}^{-1}$ ) and pressure tangential to the membrane (0.97 bars) through response surface methods, contributed to increase of flux, reaching values of about  $200 \text{ Lm}^{-2}\text{h}^{-1}$ . The accumulation of foulants caused serious problems in the function of the system but use of detergents was effective in limiting the problem.

### **6.4. Honey ultrafiltration**

Barhate et al. (2003) evaluated the effect of UF (several molecular-weight-cut-off) on

retention of enzymes from honey solution (50% H<sub>2</sub>O-50% honey) and the applicability of the procedure to protect the product from yeasts. It was found that UF membranes of 25,000, 50,000 and 100,000 molecular-weight-cut-off effectively retained amylases. Flux values ranged from 0.90 to 1.15 kgm<sup>-2</sup>h<sup>-1</sup>. It was also demonstrated that the procedure efficiently protected the product against yeasts preserving its qualitative characteristics. It was concluded that the production of i) pure honey and ii) the same product with some level of enzymes is feasible by applying combined MF and UF systems in the same procedure.

## 7. MICROFILTRATION

MF can be easily compared to UF and traditional filtration. Pores of these membranes can be manufactured from various substances. Thus, ceramic, glass, metallic or even membranes made from metal oxides, graphite or different polymers are widely used. MF can be applied in various industries either to protect products from microorganisms or for particles' separation (Han et al., 2005).

MF membranes are highly applied for carrying out solid–liquid size separation because they are characterized by several technical advantages such as moderate conditions, no phase alterations, decreased energy requirements, absence of additives, and effective design (Günther et al., 2009). The most important disadvantage of MF membranes is the existence of fouling phenomena. Specifically, at increased flow rates a layer of particles forms on the surface of the membranes. However, at decreased concentration levels no layer formation normally occurs. Another major disadvantage of this kind of membranes is the pore blocking due to accumulation

of foulants (Filippov et al., 1994).

### 7.1. Applications in the dairy industry

Flux rates, protection against microorganisms and separation in components using MF of milk were examined. Two different MF systems were applied; that is traditional and co-current permeate flow (CPF). Regarding both methods, the quantities of casein, lactose proteins and non-proteinic nitrogen content, did not differ significantly ( $P < 0.05$ ) in the product going through the membrane pores and the product retained. After the procedure, the fat concentration of the product amounted to 0.05%, while the bacterial load was limited by about 4-5 log cycles (99.84 - 99.90%). With 1% fat product, flux over a 10X rate value was about  $900 \text{ Lm}^{-2}\text{h}^{-1}$  (LMH) in the CPF system, and 400 LMH in the traditional system (Pafylas et al., 1996). Systems of MF membranes are often used by liquid products processing industries, because in addition to providing effective protection against many harmful and hazardous microorganisms, can also reduce suspended particles in liquid by improving considerably the qualitative characteristics of the product. The substantial reduction in expenditure necessary for the application of the method in recent years has made the method appealing to be applied toward meeting diverse needs (Peter-Varbanets et al., 2009). Caseins collected through MF concentration find applications in cheese production as they are characterized by greater efficiency in comparison with skim milk. Moreover, a whey product with excellent properties can be obtained from the permeation product and used as a feed during purification of serum proteinic load, with better results than cheese whey (Lawrence et al., 2008). Beolchini et al. (2004) examined the reduction possibility of

microorganisms in milk, through ongoing treatment at low temperatures (<40°C) while protecting the heat-sensitive nutrients. After a thorough analysis of membrane properties, foulants' accumulation was treated with Ultrasil25 technique that had better results than NaOH. Examinations revealed the significance of back-pulse machine to reduce fouling. Specifically, non-application of back-pulse resulted in zero permeate flux while fluxes of about 350 and 410 L h<sup>-1</sup>m<sup>-2</sup> were recorded with activation of back-pulse for 4 min and 1 min, respectively. These experiments were followed by several tests on ovine milk. Samples were initially processed with centrifugation to remove fat, and then filtered. Both centrifugation and this first filtration proved to be essential for the effective action of MF followed soon after. Permeate flux of approximately 200 L h<sup>-1</sup> m<sup>-2</sup> was observed under these circumstances (back-pulse activation at intervals of 1 min) while the method was suitable for removal of microorganisms. The permeate flux over time during milk MF under the application of two different TMP conditions is shown in figure 1.

During MF, the flux factor (J) can be described through the following equation:  $J=(1/\Omega)(dV/dt)=\Delta\Pi/\mu (R_{m,0} + R_c)$ , based on the fact that the flow resistance consists of two parameters; the resistance of the cake ( $R_c$ ) and the resistance of the membrane itself ( $R_{m,0}$ ). It is important to note that the subscript 0 is referred to the initial resistance of the membrane (Xu et al., 1995).

Furthermore, the following equation can be applied for the description of MF general properties:  $J=\Delta P/ [\mu (R_m+R_f)]$ . In this equation, J is the permeate flux, the factor  $\Delta P$  represents the TMP,  $\mu$  is the viscosity, the  $R_m$  represents the intrinsic resistance of the membrane, while  $R_f$  is the resistance caused as a result of fouling phenomena (Kwon et al., 2000).

## 7.2. Applications in fruit juice industries

Heating of products can ensure their safety, but can also cause significant losses to nutrients and sensorial properties. Because of this, MF may be valuable in the maintenance and protection of fruit juices by pathogenic and harmful microorganisms. MF is more competitive than methods of thermal treatment, because it does not require high pressures and temperatures resulting in foods' production of high safety and quality. A tubular polyethersulfone membrane ( $0.05\text{m}^2$ ) was effectively applied to clarify pineapple juice. Ten tests took place at  $25^\circ\text{C}$  (100 kPa), with the aim to evaluate the effect of MF on cold sterilization and clarification of the product. It was demonstrated that the permeate flux was slightly altered 15 min after inception. Stabilization was observed at around  $100\text{ Lh}^{-1}\text{m}^{-2}$ . Clarified products were of high quality mainly due to low haze and viscosity. Microbial examination of the process permeate that remained at  $8^\circ\text{C}$  for 28 d demonstrated that the qualitative value of the juice was in compliance with Brazilian Legislation for juices and drinks (Carneiro et al., 2002). MF procedures are commonly applied in the processing of tropical fruits since they are a key step before the production of high-added-value foods. Nevertheless, the method is not applicable to the maximum extent because the existing knowledge about the technical aspects of the treatment is limited. Since the research required for the optimization of processes is of high cost, the relevant tests are conducted on a small scale while filtrated product often returns back to the feed space to preserve a standard volumetric reduction ratio (VRR) (Vaillant et al., 2008).

### 7.2.1. Melon (*Cucumis melo* L.) juice

In the study of Vaillant et al. (2005), clarification with crossflow MF membranes followed by concentration through osmotic evaporation, was applied to melon juice. The resulting permeate quality was similar to the raw material but with smaller concentrations of suspended solid particles and carotenoids that did not penetrate the membrane. Permeate flux ranged at levels of about  $80\text{Lh}^{-1}\text{m}^{-2}$  (under conditions of continuous membrane cleaning;  $\text{VRR}=3$ ). Preservation of concentrated permeate [Total Soluble Solids (TSS) =  $550\text{ g kg}^{-1}$ ] was carried out satisfactorily. The whole procedure was effective in the production of two high-added products; a melon juice product, not deteriorating due to heat treatment, and a glowing-orange retentate with a high provitamin A concentration. The vitamin C of permeate, retentate and concentrated products was about 0.89, 0.83, 0.62 and  $0.85\text{ gkg}^{-1}$  TSS, respectively. The  $\beta$ -Carotene of the same product was about 0, 1.45 and  $0\text{ gkg}^{-1}$  TSS while glucose was determined at 157, 148 and  $162\text{ gkg}^{-1}$  TSS, respectively.

### 7.2.2. Yellow passion fruit (*Passiflora edulis* var. *flavicarpa*) juice

Passion fruit juice clarification was achieved by using liquefaction (enzymically triggered) followed by crossflow MF (membranes made of ceramics, pores of  $0.2\text{ }\mu\text{m}$ ). Increased crossflow velocity ( $7\text{ ms}^{-1}$ ) resulted in a modest reduction in fluid flow. However, the effects of enzymes on cell-wall polysaccharides, followed by MF effectively enhanced the flux rate values. It was found that combined action of pectinase and cellulase increased the permeation flux. Moreover, the combined effects of limited TMP ( $150\text{ kPa}$ ) and a large amount of enzymes ( $1\text{ ml L}^{-1}$ )

resulted in reaching the maximum degree of flux at 36°C. VRR of about 3 remained stable for 18 h, without being accompanied with limitation of flux of the filtered product, which remained almost stable at 40 L h<sup>-1</sup>m<sup>-2</sup>. The quality of permeate was effectively maintained and the only disadvantage was a relevant loss of flavor. The quality of the retentate was similar to that of the original fruit (Vaillant et al., 1999).

### 7.2.3. Pineapple

According to Carneiro et al. (2002), clarification and low-temperature sterilization of pineapple juice were carried out through crossflow MF in combination with treatment using enzymes. A tubular polyethersulfone membrane system (pores of 0.3 µm; active filtration area: 0.05 m<sup>2</sup>) was applied at room temperature (25°C) and a pressure of 100 kPa, to carry out 10 tests. The purpose of the trials was the evaluation of membrane effectiveness and its potential applicability in the treatment of this juice. Fifteen minutes after startup, the product flow passed through membrane pores, remained almost stable at 100 Lh<sup>-1</sup>m<sup>-2</sup>. Clarified products were characterized by high qualitative characteristics (low viscosity, increased transparency and stable pH, suspended particles and concentration of carbohydrates). Sterilized glass containers were used as recipients for the product and then maintained for 28 d at a temperature of about 8°C. The bottled filtered juice conformed microbiologically to the current Brazilian Legislation for juices and drinks.

### 7.2.4. Grape juice

Several experiments were conducted for testing the effectiveness of a new Filtomat® thread filtration principle for MF of semi-processed blackcurrant juice and cherry juice. The effects of many factors such as juice temperature (3-20°C), flow (20-80L/h), and pore size of the membranes (3-10µm) on the TMP, juice turbidity, nutrients, and total phenols levels were assessed. It was proved that thread MF highly limited the turbidity of the juices. As regards the blackcurrant juice, all experiments showed immediate reduction of the turbidity without affecting the protein, the sugar or the phenols concentrations. An increase in flow rates enhanced the turbidity in blackcurrant juice, but had no effect on cherry juice qualitative characteristics. It was finally demonstrated that Filtomat® thread MF can be effectively applied as a novel method for berry juice processing (Bagger-Jørgensen et al., 2002).

### 7.3. Wastewater treatment

MF is increasingly used in wastewater processing to improve its qualitative characteristics (Khemakhem et al., 2006). MF membrane made of hollow fibers (pores of 0.10 µm) was used for the retention of suspended solids from extracted yellowfin tuna spleen product. The permeate could then be processed by UF aiming at the recovery of proteases. It was demonstrated that all suspended material and about 50% of soluble protein load were effectively removed. Transmission values were around 0.8 and 0.6 for trypsin and chymotrypsin, respectively. The product that went through the membranes was yellow. It was shown that critical  $J/\tau_w$  and volume concentration factor (VCF) were related according to the gender:  $J/\tau_w = 3.29 (\text{VCF})^{-0.74}$  (at 0.15 bar). The mentioned modeling showed that there may be specific conditions under which, if not



exceeded, no accumulation of foulants is observed, while otherwise rapid increase of fouling rate takes place (Li et al., 2008).

#### **7.4. Applications in brewing industries**

Clarified beer is characterized by total absence of particles with colloidal properties and specific concentrations in macromolecular compounds such as carbohydrates, proteins, flavoring compounds, coloring etc. that determine the qualitative characteristics of the product. Gan (2001) evaluated the changes occurring during the operation of cross-flow beer MF procedure, to improve the effectiveness of membranes and produce products of high quality. Flux rate, accumulation of foulants and membrane efficiency are included among the investigated factors. Tubular ceramic membranes (Ceramem) (pore sizes: 0.1, 0.25, 0.15) were applied to carry out the process. It was demonstrated that increased flux rate was recorded by applying back-flushing. The latter was related with increased solute filtration. Continuous membrane operation without simultaneous application of back-flushing actions could drastically affect the effectiveness of the method (flux reduction higher than 95% within just 1h after starting the process) and hence the final quality of the beer.

#### **7.5. Applications in winemaking**

A gel-polarisation modeling was applied to assess various factors in the treatment of sherry wines and brandies by crossflow MF system. Many feed flows were used for the examination of

TMP effect on fluids flow. Data extracted indicate that the optimum TMP ( $TMP_{opt}$ ) and the feed flow of  $32 \times 10^{-4} \text{ l h}^{-1} \text{ Pa}$  with a  $TMP_{opt}$  of  $11 \times 10^4 \text{ Pa}$  are constant and proportional for all beverages (feed flux conditions of about  $360 \text{ l h}^{-1}$ ). D variation demonstrates that the size of the particles contained in the gel was around 10-50 nm. Furthermore, comparison of MF to conventional methods revealed that MF was much more efficient and therefore can be applied with better results. The only exception is brandy because traditional methods result in filtrated brandy of higher quality (Palacios et al., 2002). As in most cases of membranes' application, the most important disadvantage of MF during the treatment of wines is the accumulation of foulants and the reduction of membrane effectiveness or even its disablement. Before undergoing any treatment, the wine can contain a wide range of soluble and non-soluble compounds such as solute particles, different colloidal substances, various microorganisms and organic matter. Colloidal substances can be pectic, yeast forming carbohydrates and molecular aggregates as a result of the reactions of small-sized dissolved particles as wine is subjected to aging or can be caused by changes in various physical and chemical parameters or due to simultaneous contribution of the two factors. Due to the intricateness of the above parameters, it is obvious that fouling procedure can be stimulated by a variety of factors during winemaking (Vernhet & Moutounet, 2002).

## 8. OSMOTIC DISTILLATION

Osmotic distillation (OD) is a concentration method widely used in the processing industry of several of liquid foods (e.g. fruit or vegetable juices). It can effectively take place at low

temperatures, maintaining the qualitative characteristics of these perishable products. OD can be characterized as a direct osmosis, in which removal of water from a feed can occur by means of a hypertonic solution (mainly concentrated brine) flowing downstream a membrane. It can be distinguished from the common direct osmosis due to the specific membrane used, which has pores and hydrophobic properties, and is mainly fabricated from PTFE or polypropylene (Gostoli, 1999). According to Mengual et al. (1993), the volume flux of these membranes is linearly dependent on vapour pressure difference:

$J = C \Delta P_m$ . In this equation the factor  $J$  represents the volume flux/ unit of membrane surface,  $\Delta P_m$  is the water vapour pressure difference between the two sides of the membranes and  $C$  is a coefficient used for the development of the model. However, the above equation cannot be applied due to the effects of concentration-polarization phenomenon. As a result, the equation  $J = C \Delta P_m = C' \Delta P_b$  is most frequently used. In the latter,  $\Delta P_b$  is the water vapour pressure difference between concentration and pure water, and  $C'$  is the estimated or apparent osmotic distillation coefficient.

### 8.1. Application of OD on fruit products

Ali et al. (2003) examined the transfer of volatiles during the batch concentration of a sucrose solution by OD in a semi-industrial pilot plant. The transfer level of four aroma ingredients obtained from fruits was evaluated using gas chromatography. Saturation of the installation can lead to decreased adsorption level. The amount of volatiles lost is directly connected to the relative permeability rate, in comparison to water flux. The volatiles' losses

were limited significantly by applying lower circulation velocity and temperature. It was also shown that OD is characterized by lesser losses of volatiles in comparison to conventional evaporation procedures.

The effect of OD efficiency on the concentration of grape juice initially processed with UF was examined by Bailey et al. (2000). The viscosity degree of the product was measured versus concentration to extract the necessary flux measurements. UF membranes with pores' diameter of  $\leq 0.1 \mu\text{m}$  led to enhanced flux values compared to those extracted by non-ultrafiltered products. It was demonstrated that these enhanced values were due to diminished viscosity of membranes' layer after removing the protein foulants. Application of HPLC revealed that concentrated juice can be effectively obtained at decreased Brix degrees using UF process as a pre-treatment technique.

According to Barbe et al. (1998), nine different membranes, characterized by hydrophobic properties, were evaluated for their efficiency in restraining different volatile compounds during processing of their solutions with OD. After application of GC-MS and SEM, it was found that decreased volatiles flux to water flux ratios was obtained when the surface area of the membranes was larger.

The quantification of the factors implicated in concentration polarization of aquatic glucose solutions in hollow fibre molecules (PVDF fibres) during OD was studied. It was reported that the concentration polarization phenomenon significantly increased on the brine side in comparison to the feed side. The concentrations differed up to 3.17% and 1.43% on the brine and feed side, respectively, thereby leading to significant flux reduction (Bui et al., 2005).

Cassano & Drioli (2007) examined the potential use of OD for the effective concentration

of clarified kiwi-fruit juice. The experiment was carried out targeting to the minimum possible deterioration of qualitative characteristics such as ascorbic acid and TAA. It was demonstrated that at decreased levels of concentrated TSS, the evaporation flux decrease was attributed mainly to the dilution of the solution. On the other hand, at TSS levels of 35° Brix or higher, the evaporation flux is highly related to product viscosity. The application of OD did not result in any ascorbic acid losses, while evaporation through thermal processing led to a reduction level of about 87%. Similarly, the TAA remained constant after application of OD but decreased significantly (50%) after heat-treatment application.

According to Thanedgunbaworn et al. (2007), a hollow fibre membrane unit was used for the production of an OD procedure. The products processed were fructose solutions and grape juice. Several factors such as flow velocity, flux, temperature etc. were evaluated during the process. It was demonstrated that the water flux varied from 0.58 kg/m<sup>2</sup>h up to 2.02 kg/m<sup>2</sup>h, while temperature conditions and concentration of the filtered materials significantly influenced the flux. High feed and high brine velocity conditions increased the flux. High velocities were also considered responsible for the minimization of concentration polarization phenomenon. Furthermore, the temperature polarization could be significantly limited by applying increased Reynolds number and lowering the temperature.

In the study of Valdés et al. (2009), an OD procedure was used for concentrating noni juice (*Morinda citrifolia*). In this process, an OD unit based on the circulation of the products through a hollow fiber membrane (circulation degree: 0.1-1 l/min) was applied. The temperature remained constant at 30°C and under these conditions, the water flux varied within the range 0.090 and 0.413 kg/ h<sup>1</sup>m<sup>2</sup>. The concentration of the product was carried out from 8 to 32° Brix

over an hour. It was shown that the concentrations of the phenolic compounds were satisfactorily maintained.

## 8.2. Application of OD on processing of oily feeds

According to Xu et al. (2004), in contrast with conventional thermal concentration, OD can effectively maintain all nutrients (due to processing at normal temperature conditions) and decrease considerably any volatile losses that can negatively the quality of the product. Nevertheless, one of the main drawbacks of this process is the frequent wet-out of the membrane layers due to fouling. In this study, sodium alginate hydrogel was used with the aim to maintain the effectiveness of PTFE membranes. The method was found to lead to huge enhancement of adhesion strength (10-fold). Moreover, the method limited the total OD mass transfer agent by just 5%, while several tests by applying 0.2, 0.4 and 0.8 wt.% orange oil (5h) revealed that the membrane system was effectively protected (uncoated samples were wet-out rapidly when 0.2 wt.% orange oil was applied).

Mansouri & Fane (1999) evaluated the potential formation of modified membranes, characterized by hydrophobic properties and resistance against oily foulants, for carrying out OD. The conventional membranes (Celgard 25000, Millipore GVSP and UPVP) were used as patterns. PVA coatings were selected for conducting the experiment in view of their high impact on flux rates. It was demonstrated that using sucrose solutions of high concentration limited the flux degree considerably due to the development of concentration polarization phenomena. Application of coatings did not enhance the flux reduction thereby indicating that it mainly

occurred on the side of the feed supply and was not due to direct contact with membranes. The laminated layer effectively protected the membranes against fouling for a period of 24 h. Coated membranes were shown to be highly effective for carrying out OD processes, especially for processing products with high concentration in oils.

### 8.3. Cleaning of OD membranes

In the study of Gabino et al. (2007), modification of tubular ceramic membranes (macroporous alumina membranes) was carried out targeting to their application for carrying out OD procedures. The modification was based on grafting with fluoroalkylsilane, whereas the properties of the porous and the hydrophobic nature of the membranes were examined by means of SEM, mercury porosimetry, contact angle estimations and water flux rates' determination. It was shown that the above membranes could be effectively applied in the OD procedure.

The hydrophobic nature of OD membranes was evaluated during tomato puree processing and membrane cleaning procedures. The degree of hydrophobicity was estimated through measurement of penetrating drop concentration (PDC%) and penetration temperature. It was found that use of 1% NaOH displayed the highest cleaning effectiveness with regard to foulants (membrane surface tension:  $>23$  mN/m), but the hydrophobicity was lost after several fouling-cleaning efforts. It is also noteworthy that the highest cleaning effectiveness, as regards membranes with surface tension lower than 23 mN/m, was recorded for the use of P3 Ultrasil56 (Durham & Nguyen, 1994).

## 9. MEMBRANE DISTILLATION

Membrane Distillation (MD) is a thermally-based procedure used for the separation of vapour molecules which can penetrate the microporous membrane layer. The membrane is characterized by high hydrophobicity and the filtration process takes place due to vapour pressure difference (caused by temperature changes) across the membrane system. It can be applied to cover various needs such as desalination, wastewater processing, etc. and is a very useful technique for the food industry (Alkhudhiri et al., 2011). According to Lawson & Lloyd (1997) “the large vapor space required by a conventional distillation column is replaced in MD by the pore volume of a microporous membrane, which is generally on the order of 100  $\mu\text{m}$  thick. Although conventional distillation relies on high vapor velocities to provide intimate vapor-liquid contact, MD employs a hydrophobic microporous membrane to support a vapor-liquid interface”.

The mass transfer during membrane distillation procedures can be described through the following equation:  $N = - \frac{M}{\delta} (K_o K_l \Delta_{wa} \tilde{v}_G / K_l D_{wa} + x_a K_o \tilde{v}_G) \Delta_{nw}$ , where  $N$  is the mass flux,  $\tilde{v}$  is defined as the mean molecular speed of the gas,  $M$  is the molar mass,  $\delta$  is the membrane thickness,  $\Delta_{nw}$  is the water vapour mole concentration difference,  $x_a$  is the mean mole fraction of air,  $K_o$  is the factor of the Knudsen flow,  $D_{wa}$  is the ordinary diffusion coefficient and  $K_l$  is a factor related to the geometry of the membranous system (Izquierdo-Gil, 2008).

In the study of Wang et al. (2011a), a forward osmosis–MD (FO–MD) unit was firstly used to concentrate solutions of particular proteins, such as bovine serum albumin (BSA). A hydrophilic polybenzimidazole (PBI) nanofiltration hollow fiber membrane and a hydrophobic



polyvinylidene fluoride-polytetrafluoroethylene (PVDF-PTFE) hollow fiber membrane were constructed to carry out the process. It was reported that the system was characterized by high stability under particular conditions (FO dehydration rate = MD  $H_2O_{\text{vapor}}$  rate). It was also shown that the method can be effectively used for concentrating protein solutions.

### 9.1. Applications of MD on fruits and fruit juices

The comparison of OD and MD procedures, as regards the water flux rate and volatiles' (aromas: citral and ethyl butyrate) retention efficiency, was carried out by Alves & Coelho (2006). A hollow fibre membrane system was applied in the aim to concentrating a sucrose solution (fruit juice). It was demonstrated that the OD process was described by double flux rate in comparison to the MD system. This fact was attributed to the high temperature polarization impact during the operation of the MD unit. Furthermore, the MD unit displayed decreased aromas' retention efficiency when compared to OD. It was thereby found that the OD process gave better results.

Several factors such as concentration and flow rate were assessed during the application of an osmotic MD process for the treatment of phycocyanin and sweet-lime orange (Babu et al., 2006). It was shown that the flux through the membrane was enhanced from 3 up to 10 times after increasing the concentrated osmotic agent and the flow rate of phycocyanin and sweet-lime orange, respectively. The temperature polarization phenomenon was highly limited and the mass transfer was dependent on the size of the pores. To be more specific, pore size of 0.05  $\mu\text{m}$  led to increased Knudsen diffusion (74% of the total membrane resistance) whereas pore size of 0.20

$\mu\text{m}$  resulted in enhanced molecular diffusion (59% of the total membrane resistance).

Diban et al. (2009) applied MD targeting to recover ethyl 2,4-decadienoate considered the basic pear aroma substance. A hollow fiber membrane system made of polypropylene and a solution of ethyl 2,4-decadienoate in ethanol and water were applied for carrying out the experiment. It was reported that enriched aroma factors were obtained. The membrane effectively retained the volatile substance. The linear adsorption isotherm of the process can be described as,  $q_a = K_{\text{ads}} C_a^e$ , where  $K_{\text{ads}} (295.96 \text{ K}) = 0.27 \text{ kg kg}^{-1} \text{ m}^3 \text{ kg}^{-1}$ .

Jensen et al. (2011) developed a model for the prediction of the function of a direct contact MD (DCMD), during the processing of black currant juice. The system relied on the dusty gas model, used for the description of the flux through the pores of the membranes. Mass and energy principles were used and factors such as temperature and concentration polarization were assessed. For the mechanical description of the system data were taken from literature, while membrane tortuosity was calculated through several studies on pure water feed. After assessment of the procedure through experimentally obtained records, it was demonstrated that the fluxes during processing of black currant juice could be effectively estimated with an average percental error lower than 10%.

Lukanin et al. (2003) made an attempt to enhance the efficiency of MD for removing biopolymers from concentrating apple juice. Minimization of the biopolymers' percentage is feasible by applying higher enzymatic deproteinization and usage of UF for clarifying the product. The above described procedure could increase the flux rates through the membrane, during application of MD, due to the limited product viscosity which leads to lower concentration and temperature polarization effects.

## 9.2. Fouling of MD membranes

Flux degradation is basically attributed to the heat-resistant foulants. During the examination of a non-porous deposit, the flux was estimated by determining the water movement through the deposit surface. It was reported that foulants were accumulated both on the surface and among the pores of the membrane. Salt crystals developed among the pores led to structural damages. The fouling effects can be reduced by pretreating the product and carefully opting for the operating conditions of the MD process (Gryta, 2008).

## 10. MEMBRANE FOULING

According to the definition set by IUPAC “fouling is a process resulting in loss of performance of a membrane due to the deposition of suspended or dissolved substances on its external surfaces, at its pore openings or within its pores” (Madaeni et al., 2001). During membranes’ operation, fouling can occur when certain compounds in the filtered substance, are plugged or coated on membrane pores limiting the ability to filter out and disabling membranes (Jamal et al., 2004). According to Matthiasson & Sivik (1980) the concentration polarization phenomenon is one of the most severe problems occurring in the operation of membrane processes because it can cause significant decrease of the transmembrane flux. During the filtration process, a few substances in the filtered materials can be rejected by the membranes. Occasionally, some of these substances can be accumulated on the membranes’ surface and

trigger the concentration polarization phenomenon. Concentration-polarization incidents are characterized by complexity and occur when separation of substances contained in the filtered product clog membrane pores or reduce flux rate and gradually rise to qualitative degradation of the final product (Slater et al., 1985). The main problems caused by accumulation of foulants are the flux reduction and changes caused in filtered substances. Many studies and experiments around the world aimed at effectively reducing fouling and significant restoration of membranes. Nevertheless, the complete elimination of the fouling is impossible (Madaeni et al., 2001). Moreover, any research on fouling should only focus on one specific production process, as the phenomenon and its effects vary considerably per type of membrane and filtrated product (Potts et al., 1981). The significance of limiting fouling is evident. The most well known methods include pretreatment of filtrated liquids so as to minimize the amount of insoluble particles, different changes in TMP, flux and backwashing and last but not least, cleaning of the surface-pores of membranes (Madaeni et al., 2001).

According to Genkin et al. (2006), submerged hollow fibre membranes can be axially vibrated to limit fouling phenomena. It was estimated that critical fluxes of up to 60-80 l/hm<sup>2</sup> could be effectively carried out at vibrational frequencies of 10 Hz. Moreover, when transverse vibrations were added, the critical flux was doubled to 130 l/hm<sup>2</sup> at the same vibrational frequencies. Use of coagulants can significantly increase the critical flux values, especially at decreased vibrational frequencies. Particularly, at a frequency value of 1.7 Hz the critical value was enhanced from 17 to 46 l/hm<sup>2</sup>. After combination of axial and transverse vibrations, the critical flux increased by up to 5 times (with addition of 34 mg/l aluminium chlorhydrate). At higher frequencies, the critical flux started to decrease. The latter was attributed to floc breakup

due to turbulence enhancement. It was shown that the desirable critical flux values could occur at low frequency vibration in the presence of an appropriate coagulant.

Engler & Wiesner (2000) studied the effects of different factors, such as rotation rate, TMP and feed suspension, of rotating disk membranes on the development of fouling. It was demonstrated that the permeate flux was slightly affected by feed steam concentration. Fouling degree was limited by increasing rotation rate and enhanced by increasing TMP. Increased rotation speeds led to significant decrease of fouling.

Polyphenolic substances can easily lead to formation of sizeable particles through binding of proteins and peptides. The latter are usually responsible for plugging membranes. It was shown that dimers and trimers of catechin and epicatechin can be bound with proteins and peptides in beer products. Furthermore, it was demonstrated that brewing of beer using decreased levels of proanthocyanidin malt can result in better diatomaceous earth filtration function (Stewart et al., 1998).

According to Fang & Shi (2005), “pore fouling is mostly caused by the deposition of colloidal matters inside the membrane porous structure and the formation of a cake layer on the membrane surface, resulting in the increase of filtration resistance. Filtration resistance  $R$  ( $\text{m}^{-1}$ ) may be expressed, according to Darcy’s law, by the following equation:  $R = \Delta P / \mu J$ , where  $\Delta P$  is the trans-membrane pressure gradient ( $\text{N m}^{-2}$ ),  $\mu$  the viscosity of the permeate ( $\text{N s m}^{-2}$ ), and  $J$  the permeation flux ( $\text{m s}^{-1}$ ). For the filtration of activated sludge, the total permeation resistance,  $R_t$ , may be expressed as the sum of three components:  $R_t = R_m + R_p + R_c$ , where  $R_m$  is the intrinsic membrane resistance,  $R_p$  the pore fouling resistance, and  $R_c$  the cake layer resistance.”

Hu et al. (2004) evaluated the separation of water from oil-in-water emulsion using a UF

system at several TMPs. Several factors such as intrinsic and gel resistance were estimated. It was found that numerous membrane properties, feed emulsion concentration and temperature can greatly affect the fluctuations of permeate flux at different TMPs. By enhancing the pressure, the flux increased at decreased emulsion concentration, while at increased emulsion concentration conditions, the occurrence of concentration polarization phenomenon proved to be a serious issue.

### 10.1. Fouling of ED membranes

According to Ruiz et al. (2007), one of the main problems in the use of ED membrane systems is the accumulation of foulants and the gradual decrease in the efficiency of ion-exchange membranes. Pulsed power application (application of non-changing current density for a given period of time ( $T_{on}$ ) accompanied by a current interruption time) was shown to be effectively used to reduce fouling. The same authors evaluated the effect of pulsed electric field (PEF) ( $T_{on}=10s-T_{off}=10s$ ,  $T_{on}=10s-T_{off}=40s$ ) compared with dc current after ED application on a casein solution at a current of 10, 20 and 30 mA/cm<sup>2</sup>. It was found that under these circumstances, PEF could preserve membranes from foulants' accumulation. Based on the obtained data it was concluded that within the first 10s–40s (PFE state), no accumulation of foulants was observed on any of the cases, while for 10s–10s PEF fouling appeared only over 10 mA cm<sup>-2</sup>. Foulants mainly consisted of proteins (97%) and were generated due to the increase of dissociated molecular H<sub>2</sub>O or temperature conditions near the membranes. The period of current pause can reduce the extent of these events.

Wang et al. (2011b) applied bipolar membrane electrodialysis (BMED) to regenerate NH<sub>4</sub><sup>+</sup>

and  $\text{H}_2\text{SO}_4$  originating from effluents glutamate production industries. Fouling was detected during the application of the method. An examination was carried out to detect the substances from which foulants of cation-exchange membrane systems (CEMs) are consisted and evaluate the effectiveness of different technologies in addressing fouling. After treatment of 5 BMED batches, SEM was used to detect a large proportion of foulants on CEM outer surface layer. Moreover, the degradation of various physicochemical factors led to the conclusion that there was also increased concentration of foulants in the inner membrane layers. After conducting the appropriate analysis, the foulants accumulated in the outer layers of the membrane were shown to consist of  $\text{Ca}(\text{OH})_2$ ,  $\text{CaCO}_3$  and  $\text{Mg}(\text{OH})_2$ . Application of acid cleaners combined with ultrasound implementation proved to be the most appropriate way for disposing membranes of foulants and restoring them to their original operating condition.

## 10.2. Fouling of RO membranes

RO membrane fouling can be caused by three different groups of constituents; slightly soluble non-organic substances (e.g.  $\text{CaSO}_4$ ), colloidal compounds or free particles and dissolved organic substances (Potts et al., 1981). When RO membranes are employed toward processing milk and other dairy products, the intensity of fouling increases considerably. Specific models were used to assess the effect of these two factors in the fouling for a tubular RO pilot-plant applied to the concentration of cheese whey (period covered: 20h). The factors taken into account during the carrying out of the experiment were; pressure conditions at the start of the process, flux rate, disposal rate of concentrated product and temperature conditions. It was

shown that the description pattern of gel layer fouling is not effectively correlated with the evaluated accumulation of foulants. However, a precipitation model for  $\text{Ca}(\text{H}_2\text{PO}_4)_2$  confirmed the experimental data (Boxtel et al., 1991). The RO membrane cleaning processes could be classified into 2 main groups: physical and chemical. The simultaneous application of both techniques does occur in some cases. The concept of physical cleansing may contain sponge ball cleaning, correction of the problem by increasing flow pressure or osmotic backwashing. Sponge ball method has the disadvantage of tubular structure demand and can be exclusively applied when turbulent promotive systems and any other factors that impede its smooth functioning, are not used (Potts et al., 1981). Cleaning RO membranes with chemical methods may include the use of acids, bases, active surface cleaners and detergents. The rate and efficiency of cleaning are directly correlated to the type of detergent and the total amount used. The cleaning action is enhanced by increasing the proportion of applied cleaning agent (Madaeni et al., 2001).

Fouling phenomena due to inorganic scaling represent severe problems that limit considerably the use of RO technique in several water process operations. Torsional vibration of flat sheet membranes can be applied with the aim to reducing fouling. It is mainly based on inducement of enhanced shear rates at the membrane surface. The development of fouling in RO membranes was assessed with the use of a vibratory shear enhanced filtration process (VSEP, New Logic Research, Emeryville, CA) unit, applied for the treatment of a simulated brackish water source and brine. Application of vibration limited significantly the development of fouling and effectively augmented the water recovery from 80% to 90%, as regards the brackish solution, and from 50% to 75% in the case of brine. During vibration, fouling was highly limited in all cases. Moreover, vibration was effectively used to enhance the rejection of major ions in



both the brackish solution (from 70%-88% to higher than 95%) and the brine (from 70%-88% to higher than 90%) (Shi & Benjamin, 2009).

In the study of Lee & Lueptow (2003) the application of rotating RO, which takes advantage of Taylor-Couette flow instabilities to limit concentration polarization phenomenon and fouling, was assessed for its effectiveness on controlling the  $\text{CaSO}_4$  scale formation. According to the same authors “The permeate flux for rotating RO at  $\omega = 180\text{rpm}$  remains constant up to a volume concentration factor (VCF) of 4.2, while the permeate flux declines steadily with increasing VCF for no rotation”. This is attributed to the fact that vortices in rotating RO cause bulk crystallization and inhibit scale particle accumulation on the surface of the membranes. The anti-scaling property can be enhanced by increasing rotational speed and is partially dependent on TMP.

### **10.3. Fouling of UF membranes**

The main disadvantage during the application of UF membranes is the gradual flux reduction due to increased solute polarization around membrane surface layers and the accumulation of foulants due to deposition of various macromolecular compounds and colloidal substances (De Bruijn et al., 2005). Besides the above, UF membrane fouling can be referred to the growth and multiplication of different microorganisms and the covering membrane surface with various materials thereby resulting in flux decline. According to Kazemimoghadam & Mohammadi (2007) cleaning can include the use of physical, chemical and biological cleaners. De Bruijn et al. (2002) reported that accumulation of foulants is usually not repairable and leads

to a gradual reduction of permeate flux. Where it is possible to restore the films in their original condition, the problem has been caused by coating membrane layer with colloidal and particles of the filtered substance. This trend may result in concentration polarization incidents and can be resolved with cleaning of membrane surfaces. However, when fouling is not repairable, these substances penetrated inside membranes thus blocking the membrane pores and turning the membrane into obsolete.

#### **10.4. Fouling of MF membranes**

During operation of MF membranes, fouling can take place through accumulation of proteins or other foulants of biological origin, on surfaces of membrane systems especially with application of high flow (Wakeman & Williams, 2002). The gradual inevitable emergence of fouling phenomenon reveals that in real conditions, the problem cannot be fully eliminated. As a result, the required system operating expenses increase because of frequent use of cleaning agents to restore the membranes' effectiveness. The cleaning frequency of membranes as well as the method selected in each case depend both on the processed product and membrane properties (construction material-resistance to chemicals). The simultaneous use of physical and chemical methods is common and can be applied for optimum cleaning of membrane surfaces (Al-Malack & Anderson, 1997). Therefore, cleaning is a major parameter for the proper functioning of membrane systems. Improvement of cleaning agents includes optimization of effectiveness, practical use and, preferably, immediate action whereas the effects of substances on membranes and equipment, and compliance with national and international safety and quality standards are

of crucial importance as well (Makardij et al., 1999). Backpulsing is widely applied nowadays to clean membranes from accumulated foulants. The method demands high-frequently reversed flux permeation and the driving force of the method is the application of pressure to the product that has already passed through the membrane, so as to move in the opposite direction (Günther et al., 2009).

### 10.5. Fouling of NF membranes

According to experiments conducted by several researchers, fouling of NF membranes is mainly due to protein substances and alters lactic acid fermentation broth filtration. Moreover, the presence of several ions such as supersaturated calcium salts in the application of membranes in milk processing industries, can gradually affect the functionality of these membranes. As a result, there is an urgent need for more studies on the accumulation of foulants, membrane function and the effect of calcium on the system (Jeantet et al., 1996). The application of membranes during processing dairy products occurs at temperatures that restrict the growth of most bacteria (at temperatures lower than 10°C or higher than 50°C). Functionality of membranes, their progressive deterioration and infiltrated product properties have not been fully investigated during the implementation of various other temperatures. It was demonstrated that during cheese whey UF process, the flux factor was limited by enhancing temperature conditions by 20°C (from 10°C to 30°C). The fact was attributed to the limited percentages of  $\text{Ca}(\text{H}_2\text{PO}_4)_2$  presented at these temperatures. Moreover, at temperatures higher than 30°C a flux increase was

observed. This increase was due to low viscosity and high diffusivity conditions, that overshadowed the effect of  $\text{Ca}(\text{H}_2\text{PO}_4)_2$  (Rice et al., 2009b).

## 11. CONCLUSIONS

Membrane Processes (MPs) have found a continuously increasing number of applications in the protein industry (whey/milk protein standardization/concentration, soy protein isolates), alcoholic and non-alcoholic drinks (wine, beer, fruit juice cloudy and clear) and egg products over the last 35 years. MPs have a crucial advantage over other technologies; that is they can potentially support sustainable industrial growth by saving energy, decreasing capital costs, lessening environmental impact, and optimizing the exploitation of raw materials. MPs have already replaced their conventional energy intensive techniques thus leading to substantial drop in energy, cost and environmental impact (van Gerven and Stankiewicz, 2009). Electrodialysis (ED) with bipolar membranes (BMED) has been effectively used to treat/separate food products such as fruit juice, soybean proteins and wines with regard to purity. The main disadvantages of this technique are its being prone to pores blocking and the relatively high cost of equipment (Bazinet et al., 1998). The former can be effectively resolved by including a centrifugation step. Microfiltration is effectively applied to make sure the microbiological limpidity and stability of wines. Combination of ED with Cross flow MF was found to be advantageous compared to each technique individually because it can provide solution to the problems of microbiological stability, clarification, tartaric stabilization and oxidation of wine (Daufin et al., 2001). Reverse Osmosis (RO) desalination plants are the top leaders in desalination because RO installations stand for 60% against 34.8% for the thermal process of installation plants worldwide. The

greatest advantages of RO over other technologies are the following; i) less carbon dioxide released and ii) less concentrated brine to be disposed as waste.

As regards the future trends and perspectives, membrane distillation (MD) is a novel appealing technology depending on hydrophobic membranes for the separation of aqueous solutions. MD is an alternative to RO and evaporation. The main advantages of MD are its 100% efficiency to reject ions, non-volatile components and macromolecules, application of lower temperatures than its conventional competitors, less membrane fouling, and lower operating pressure across the membrane (Drioli et al., 2011). It is currently believed that most MPs can further enhance their effectiveness by applying integrated membrane systems similarly to the pre-treatment in the case of RO. The combination of integrated distillation-pervaporation systems has considerable advantages such as lower energy consumption (50-60%), enhanced product quality and wider application range of this technology making it capable of testing azeotropic mixtures (Lipnizki et al., 1999).

As a concluding remark one can say that membrane science and technology offer a great range of options in terms of design and optimization of innovative production. The latter can be considerably enhanced if emphasis is put on integrated membrane operations, which can prove very effective (less energy consumption and lower environmental impact) in agro-food and petrochemical industry, biotechnological processes and gaseous or aqueous mixtures separations.

## 12. ABBREVIATIONS

AFM          Atomic force microscope

|      |                                     |
|------|-------------------------------------|
| BMED | Bipolar membrane<br>electrodialysis |
| BOD  | Biochemical oxygen demand           |
| CEM  | Cation exchange membrane            |
| COD  | Carbon oxygen demand                |
| COD  | Chemical oxygen demand              |
| CPF  | Current permeate flow               |
| DDT  | Dichlorodiphenyltrichloroethane     |
| DGP  | Distilled grape pomace              |
| ED   | Electrodialysis                     |
| EDBM | ED with bipolar membranes           |
| HF   | Hollow fibre                        |
| IE   | Ion exchange                        |

|        |  |
|--------|--|
| MF     | Microfiltration                            |
| MPT    | Membrane Processing<br>Technology          |
| MUP    | Whey and milk ultrafiltration<br>permeate  |
| NF     | Nanofiltration                             |
| NOM    | Natural organic matter                     |
| OWRT   | Office of Water Research and<br>Technology |
| PEEKWC | Modified poly(ether ether<br>ketone)       |
| PEF    | Pulsed electric field                      |
| PES    | Polyethersulfone                           |
| PPO    | Polyphenylene oxide                        |
| PSU    | Polysulphone                               |
| PVC    | Polyvinyl chloride                         |

|                |                              |
|----------------|------------------------------|
| PVDF           | Polyvinylidene fluoride      |
| R <sub>m</sub> | Resistance factor            |
| RO             | Reverse osmosis              |
| SEM            | Scanning electron microscopy |
| SOCs           | Synthetic organic chemicals  |
| TAA            | Total antioxidant activity   |
| TMP            | Transmembrane pressure       |
| TOC            | Total organic carbon         |
| VCF            | Volume concentration factor  |
| VRR            | Volumetric reduction rate    |



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## 14. TABLES

**Table 1:** Representative examples of Electrodialysis applications on processing of several food products: purpose, membrane materials, conditions and evaluation of filtration.

| <b>Filtration target</b> | <b>Membrane material and/or type</b>   | <b>Filtration conditions (under pressure, temperature, etc.)</b> | <b>Filtered products</b>  | <b>References</b>        |
|--------------------------|--|--|---|--------------------------|
| Demineralization         | Monovalent ion selective membrane  | 24°C   | Skim milk (K <sup>+</sup> , Na <sup>+</sup> , Cl <sup>-</sup> , Ca <sup>2+</sup> , Mg <sup>2+</sup> , PO <sup>3-</sup> <sub>4</sub> ) | Andrés et al. 1995       |
|                          | Prototype laboratory electrodialyzer with ion-exchange membranes   | 35°C   | Vinasse   | Milewski & Lewicki, 1988 |
|                          | Electrodialyzer (P1; EIVS, Le Vésinet, France) consisting of 20 cells with alternating CMV cation and AMV anion exchange membranes (Asahi Glass Corporation, Tokyo, Japan) | 20 ± 2°C, pH: 7.4–7.6  | Mussel cooking juices   | Cros et al., 2005        |



|   |  |   |  |                            |
|---|--|---|--|----------------------------|
| Stabilization   | Polymeric selective permeable membranes  | Deionisation rate value: higher than 26% in the Fino wine, and lower than 20% in Medium and Cream wines | Sherry wines (“Fino”, “Medium” and “Cream”)      | Benítez et al., 2003       |
| Stabilization - potassium hydrogen tartrate (KHT) removal | EURODIA EUR2C-7P18 electrodialyser   | 25°C, 0.2 g/l NaCl solution,  | Wine tartaric (wine and red “Vinho Verde” wines) | Gonçalves et al., 2003     |
| Study the variation of the acid-salt equilibrium          | Membranes (CMX/AMX Neosepta obtained from Ameridia Division of Eurodia SA  | pH 7, NaOH (Merck)  | Citric acid                                      | Elías-Serrano et al., 2007 |
| Separation - Citrate recovery                             | Laboratory-scale electrodialyser (Aqualyzer PI, Corning EIVS, Le Vesinet, F)   | 33°C, pH: 3-7   | Citric acid in aqueous solutions                 | Moresi & Sappino, 1998     |
| Separation - Lactic Acid recovery                         | Electrodialyzer: 10-compartment cell stack (TS-1-10; Tokuyama, Tokyo), a DC power supply (PE 1646; Phillips, Brussels, | 0–40°C, pH: 0–10  | Lactic acid solution                             | Wee et al., 2005           |

|  |   |                                     |  |                   |
|--|---|-------------------------------------|--|-------------------|
|  | Belgium), three magnet pumps (MD-15R; Iwaki, Tokyo), and three holding tanks made of acrylic resin. |                                     |  |                   |
|  | ED with bipolar membranes and two compartments (EDBM2C configuration)                               | Initial pulpy juice, pH: 4.5, 25 °C | Passion fruit juice ( <i>Passiflora edulis</i> v. <i>flavicarpa</i> , Degener) | Vera et al., 2009 |

**Table 2:** Representative examples of Microfiltration applications on processing of several food products: purpose, membrane materials, conditions and evaluation of filtration.

| <b>Filtration target</b>                | <b>Membrane material and/or type</b>   | <b>Filtration conditions<br/>(under pressure, temperature, etc.)</b> | <b>Filtered products</b> | <b>References</b>      |
|---|--|--|--------------------------|------------------------|
| Reduction of microbial content          | Tangential flow laboratory pilot plant Membralox <sup>®</sup> XLAB3 (EXEKIA, Bazet - France), with a single tube ceramic membrane Membralox <sup>®</sup> T1-70 (pore diameter 1.4µm) | ≤40°C, pH: 6.41-6.5  | Bovine and ovine milk    | Beolchini et al., 2004 |
| Reduction of microbial content          | Membralox IPI9-40  | 50°C, 15 psi (1.03 bar)  | Milk                     | Pafylas et al., 1996   |
| Clarification - reduction of pollutants | Plastic pastes were prepared from ceramic powder of clay (SI2) mixed with organic additives and water  | 250°C and 1080°C   | Cuttlefish effluents     | Khemakhem et al., 2006 |

|                                      |  |  |                     |                       |
|--------------------------------------|--|--|---------------------|-----------------------|
| Clarification                        | Pellicon Cassette unit from Millipore (Bedford, USA).<br>Twenty microporous membrane films of polyvinylidene difluoride (PVDF)           | 4 to 50 °C,<br>11X10 <sup>4</sup> Pa                           | Sherry wines        | Palacios et al., 2002 |
|                                      | Two tubular ceramic multichannel membranes (Membralox IP19-40, SCT, Bazet, France)   | 36°C, 150 kPa, high enzyme concentration (1 mL <sup>-1</sup> ) | Passion fruit juice | Vaillant et al., 1999 |
| Cold sterilization and clarification | Tubular polyethersulfone 0.3 µm pore size membrane.  | 25°C, 100 kPa,   | Pineapple juice     | Carneiro et al., 2002 |
| Fractionation and Clarification      | Tubular ceramic membranes (Ceramem) with nominal pore diameters of 0.2, 0.5, and 1.3µm   | 2.0 ± 0.9°C,   | Beer                | Gan, 2001             |
| Separation of components             | PVDF (polyvinylidene difluoride) membrane made in USA by Desal Desalination Systems Inc, type JX, with an average pore diameter of 0.3mm | 90°C, 70 bar, pH: 2-11   | Milk                | Makardij et al., 1999 |
| Production of a casein               | 0.3µm (FG) or 0.5µm (FH) polyvinylidene fluoride   | ±3°C, 170 to 30kPa   | Skim milk           | Lawrence et al., 2008 |

|                                |  |                        |                                  |                 |
|--------------------------------|--|------------------------|----------------------------------|-----------------|
| concentrate                    | (PVDF) membrane (PTI Advanced Filtration, California, USA)                               |                        |                                  |                 |
| Removal of suspended particles | Hollow fiber membrane (CFP-1-E-5A, Amersham Biosciences, UK) (pore size of 0.10 $\mu$ m) | 28 $\pm$ 2°C, 0.15 bar | Extract of yellowfin tuna spleen | Li et al., 2008 |

**Table 3:** Representative examples of Nanofiltration applications on processing of several food products: purpose, membrane materials, conditions and evaluation of filtration.

| <b>Filtration target</b>                    | <b>Membrane material and/or type</b>  | <b>Filtration conditions (under pressure, temperature, etc.)</b> | <b>Filtered products</b>            | <b>References</b>      |
|---|---|--|-------------------------------------|------------------------|
| Concentration and Purification              | Thin-film composite Desal-5 membrane, model DK2540, produced and supplied by Osmonics         | 20°C±1°C, pH: 6.62, 0 bar  | Tomato industry wastewater effluent | Iaquinta et al., 2009  |
| Purification of lactic acid and amino acids | Organic NF membranes PES10 and N30F (purchased from Nadir, Germany) and MPF36 (Koch, Germany) | 22°C, 3.5 MPa  | Silage juice                        | Koschuh et al., 2005   |
| Concentration                               | YPROLAB-2 (made by Millipore Co)  | 30–50°C, 10–20 bar   | Milk, whey proteins                 | Atra et al., 2005      |
|   | XN45 (Trisep) membrane  | 30–50°C, 20 bar  | Red wine                            | Banvolgyi et al., 2006 |

|                          |   |  |  |                           |
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| Recovery of lactic acid  | MSP 006239 Prolab System (Millipore, Molsheim, France) equipped with a R76A Millipore NF membrane (spiral wound module) | 42°C, pH: 6.0, NF membranes coupled with a CSTR  | Whey   | Jeantet et al., 1996      |
| Recovery of antioxidants | Nanomax 95, Nanomax 50 (PA/PS) and Inside Céram (ceramic membrane)  | 20°C, pressure: Millipore membranes, 8bar; Inside Céram membrane, 6 bar                        | Industrial waste liquors                               | Díaz-Reinoso et al., 2010 |
| Fractionation            | PA/PS   | 20 ± 4°C, 2–8 bar  | Aqueous extracts from distilled fermented grape pomace | Díaz-Reinoso et al., 2009 |
| Treatment                | Polymeric materials such as polysulfone and polyamide.  | 30°C, pressure: 10 to 30 bars, previous filtration through a 10 and 5y cartridge safety filter | Sugar decolorizing resin regeneration waste            | Cartier et al., 1997      |
|                          | Polyamide circular flat membrane Desal 5 DK (Osmonics, USA)   | 45°C, 4000 kPa   | Dairy process waters                                   | Frappart et al., 2006     |

|                  |  |  |  |                            |
|------------------|--|--|--|----------------------------|
|                  | Polyamide thin-film-composites (TFC)   | 25+1°C, pH: 2-11, pressure: 0.483 - 2.758MPa | Water solutions containing humic acids | Wang et al., 2005          |
|                  | Polyethersulfone   | 8 bar  | Whey                                   | Yorgun et al., 2008        |
| Dealcoholization | NF-HL, NF-DK   | 5 - 20 bar                                   | Wine                                   | García-Martín et al., 2009 |
| Demineralization | NF40 (FILMTEC)   | 45°C, pH: 2-11, pressure: 2.5-40 bar         | Whey products                          | Van der Horst et al., 1995 |
|                  | Aromatic polyamide spiral-wound membrane. Osmonics (USA) (model DK2540C)   | 2.0 - 4.0MPa, pH: 6.2-6.8                    | Whey and milk ultrafiltration permeate | Suárez et al., 2006        |
| Desalination     | NF270, NF-, NF90 (Filmtec/DOW), Desal-5 DL (GE Osmonics) (Polyamide, Polypiperazine amide, Polyamide, Polyamide) | 22°C, 35bar                                  | Soy sauce                              | Luo et al., 2009           |



|  |  |  |                          |                           |
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|  | Polyamide thin-film composite membranes                | 10°C, pH: 5.5, ultrafiltered product         | Dairies                  | Rice et al., 2009b        |
|  | Ceramic, tubular (US Filter)                           | Room temperature, pH: $8.5 \pm 0.5$ , 690kPa | NaCl solution            | Skluzacek et al., 2007    |
|  | Thin-film DS5 membrane from Desalination Systems (USA) | 50°C, 5-28 bar, pH: 2-11                     | Salt and sugar solutions | Vellenga & Trägårdh, 1998 |

**Table 4:** Representative examples of Reverse Osmosis applications on processing of several food products: purpose, membrane materials, conditions and evaluation of filtration.

| <b>Filtration target</b> | <b>Membrane material and/or type</b>                      | <b>Filtration conditions (under pressure, temperature, etc.)</b> | <b>Filtered products</b>                     | <b>References</b>      |
|--------------------------|---|--|--|------------------------|
| Concentration            | Polyamide tubular membranes                               | 5.50MPa  | Apple juice                                  | Alvarez et al., 1997   |
|                          | Spiral wound aromatic polyamide membrane                  | 25°C, TMP: 1.5–3.5MPa  |  | Álvarez et al., 1998   |
|                          | Polyamide flatsheet RO membrane from Trisep               | 30°C, TMP: 30-50 bar, ultrafiltered juice was used               | Blackcurrant ( <i>Ribes nigrum</i> L.) juice | Bánvölgyi et al., 2009 |
|                          | Tubular thin-film composite reverse osmosis (RO) membrane | 30°C and 50°C, 2.1-2.8MPa  | Milk   | Cheryan et al., 1990   |
|                          | Filtration unit MicroLab 40S (VMA Meung, France)          | 40°C, 50 bar, the product was                                    | Mussel cooking juices:                       | Cros et al., 2005      |

|               |   |   |  |                                |
|---------------|---|---|--|--------------------------------|
|               |   | previously<br>desalinated by<br>ED  |  |                                |
| Concentration | Aromatic polyamide spiral-wound one from Dow Chemical (Filmtec SW30-2540).  | 21-39°C,<br>applied<br>pressure: 0-55<br>bar,   | Waste<br>leaching liquid<br>from the citric<br>juice<br>production<br>industry | García et<br>al., 2002         |
|               | Lab Unit 20 supplied by DSS (Denmark). A plate and frame module composed of HR98PP thin film composite membranes (DSS, Denmark) | 20, 30 and<br>40°C, TMP:<br>40, 50 and 60<br>bar (best<br>process<br>conditions:<br>40°C, 60 bar) | Grape juice  | Gurak et<br>al., 2010          |
|               | MFT-Köln RO type  | 40°C, 40 bar  |  | Rektor et<br>al., 2007         |
|               | Polysulphone/polyethylene composite layer membranes (95% NaCl rejection) from DSS (HR98PP model)                                | 25°C, TMP:<br>20, 40 and 60<br>bar  | Orange ( <i>Citrus sinensis</i> ) juice  | Jesus et al.,<br>2007          |
| Concentration | The BW30 RO membrane (DOW-FILMTEC) made of polyethylene terephthalate and bisphenol A polysulfone                               | 30 - 45°C,<br>TMP: 22 bar   | Thin sugar<br>juice  | Madaeni &<br>Zereshki,<br>2008 |

|                                  |   |  |  |                                 |
|----------------------------------|---|--|--|---------------------------------|
|                                  | LAB UNIT M-20—DDS<br>with thin film composite<br>membranes  | 25°C, TMP:<br>6MPa,<br>Preclarified<br>juice | Acerola juice  | Matta et<br>al., 2004           |
|                                  | AFC-80 polyamide tubular<br>membrane  | 25°C, 60 bar                                 | Blackcurrant<br>juice                                | Pap et al.,<br>2009             |
| Concentration                    | Aromatic polyamide<br>membrane HR 95 PP of<br>95% NaCl rejection and HR<br>98 PP of 97.5% NaCl<br>rejection | 0-60°C, 0-<br>60bar                          | Ester and<br>aldehyde<br>solutions                   | Pozderović<br>et al., 2006      |
|                                  | Composite membrane (95%<br>NaCl rejection)  | 20–35°C,                                     | Camu–camu<br>juice<br>( <i>Myrciaria<br/>dubia</i> ) | Rodrigues<br>et al., 2004       |
|                                  | Daicel's cellulose acetate<br>RO membranes (Daicel<br>Chemical Industries Ltd,<br>Japan)                    | 35°C, 60 bar                                 | Tomato<br>concentrates                               | Yildiz et<br>al., 1993          |
| Dealcoholization                 | Spiral-wound system   | 0°C, 35 - 50<br>bar                          | Homemade<br>alcoholic<br>beverages                   | Pilipovik &<br>Riverol,<br>2005 |
| Retention of<br>carboxylic acids | Aromatic polyamide<br>membrane (Toray: TR70-<br>2514 F)   | 18 to 23°C, 10<br>bar                        | Waste water  | Tödtheide<br>et al., 1997       |

|           |  |              |   |                     |
|-----------|--|--------------|---|---------------------|
| Treatment | RO spiral-wound membrane with polyamide active layer | 25°C, 20 bar | Dairy industry wastewater (mixtures of milk, whey and cream with dry matter ranging from 0.4 g.L <sup>-1</sup> to 71 g.L <sup>-1</sup> , fat content (0 to 22%) and heat treatment) | Vourch et al., 2008 |
|-----------|--|--------------|---|---------------------|

**Table 5:** Representative examples of Ultrafiltration applications on processing of several food products: purpose, membrane materials, conditions and evaluation of filtration.

| <b>Filtration target</b>                                  | <b>Membrane material and/or type</b>                                    | <b>Filtration conditions (under pressure, temperature, etc.)</b> | <b>Filtered products</b> | <b>References</b>     |
|---|---|--|--------------------------|-----------------------|
| Fractionation   | Tubular ceramic membrane  | 30°C, TMP: 1 bar, pH 3, 9 and 10, clarified whey was used        | Whey proteins            | Almécija et al., 2007 |
| Rejection of enzymes                                      | Polytetrafluoroethylene membranes                                       | 25°C, 1.0MPa   | Honey                    | Barhate et al., 2003  |
| Rejection of polyphenols and brown colour (discoloration) | Membranes made of polyethersulfone (PES) and polyvinylpyrrolidone (PVP) | 1 bar  | Apple juices             | Borneman et al., 1997 |

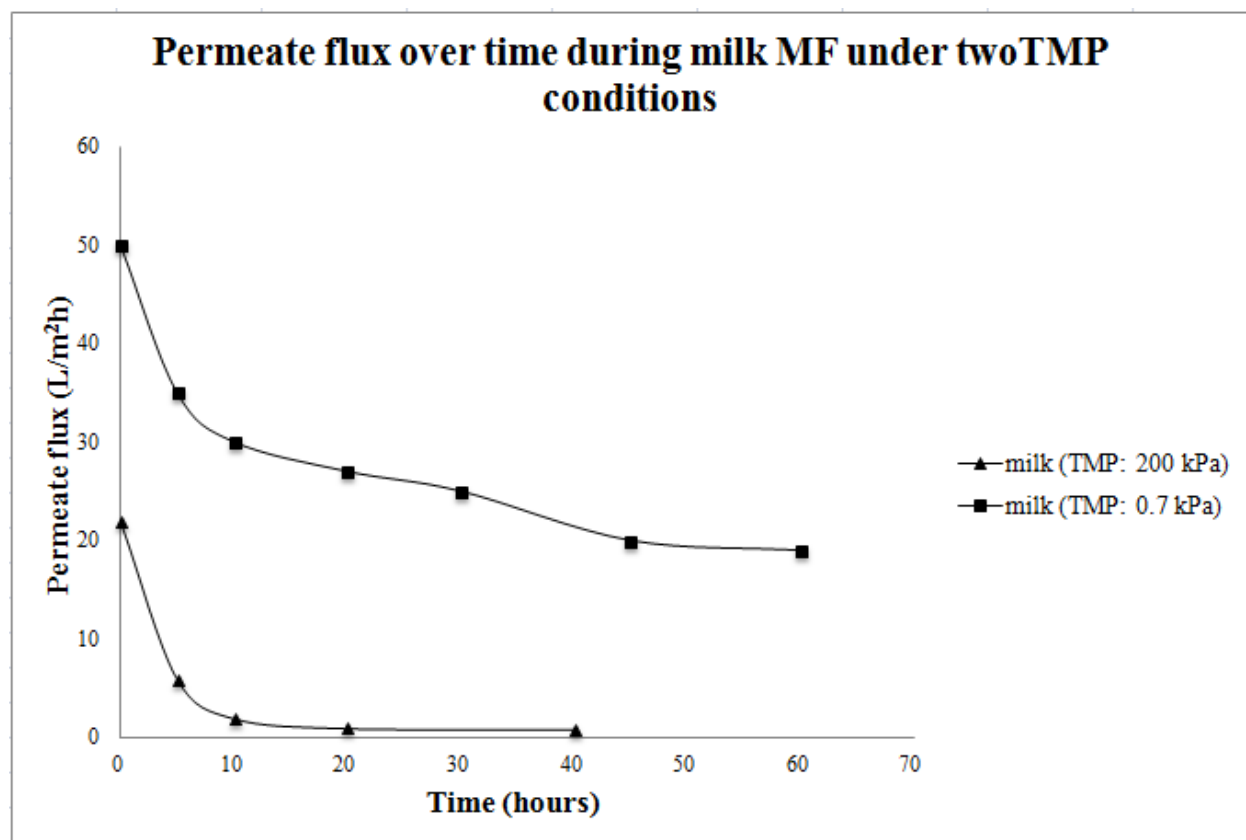
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| Treatment     | Biomax <sup>TM</sup> membrane made of polyethersulfon   | 20°C, TMP: 1.8 bar  | Cork processing wastewaters | Benítez et al., 2006     |
| Clarification | Zirconium oxide (15 kDa, 50 kDa) membranes  | 50-55°C, 400 kPa (optimal)  | Apple juice                 | De Bruijn et al., 2002   |
|               | Carbosep <sup>R</sup> tubular inorganic membrane  | 50-55°C, TMP of 150 and 400 kPa   |                             | De Bruijn et al., 2003   |
|               | Millipore Corp, Bedford, USA made of polyethersulfone (PES) and Millipore Corp, Bedford, USA made of cellulose acetate (CA) | Roome temperature, $1 \cdot 10^5$ , $2 \cdot 10^5$ , $3 \cdot 10^5$ and $4 \cdot 10^5$ Pa |                             | Gökmen & Çetinkaya, 2007 |
|               | Tubular polyvinylidene fluoride (PVDF) membranes  | 25°C, TMP: 0.85 bar, pH: 2-11   | Blood orange juice          | Cassano et al., 2007b    |
| Clarification | Tubular membrane module (Koch Series-Cor <sup>TM</sup> HFM 251, polyvinylidene fluoride)                                    | 25°C, TMP: 90kPa  | Kiwifruit juice             | Cassano et al., 2007a    |
|               | Modified poly(ether ether ketone) hollow fibre membranes  | 15-35°C, 0-125kPa   |                             | Tasselli et al., 2007    |

|                            |   |   |                           |                            |
|----------------------------|---|---|---------------------------|----------------------------|
|                            | Modified poly(ether ether ketone) (PEEKWC) and polysulphone (PSU) hollow fibre (HF) membranes   | 25±2°C, TMP: 0.3 bar                                      | Clementine mandarin juice | Cassano et al., 2009       |
| Clarification              | Fluoropolymer (FS50PP) and regenerated cellulose (MWCO) membranes   | 50°C, TMP: 0.5 bar, product reconstituted from tea powder | Hot tea extract           | Evans & Bird, 2006         |
|                            | Ceramic membrane  | 25°C, 300kPa  | Pineapple juice           | Jiraratananon et al., 1997 |
| Clarification/purification | UF-PES-030 (30kD, permanently hydrophilized PES), UF-PES-050H (50kD, permanently hydrophilized PES), UF-PS-100H (100kD, permanently hydrophilized PS), GR51PP (100kD, PS) and GR40PP (50kD, PS) | 22–25°C, 1 bar  | Sugarcane juice           | Saha et al., 2006          |

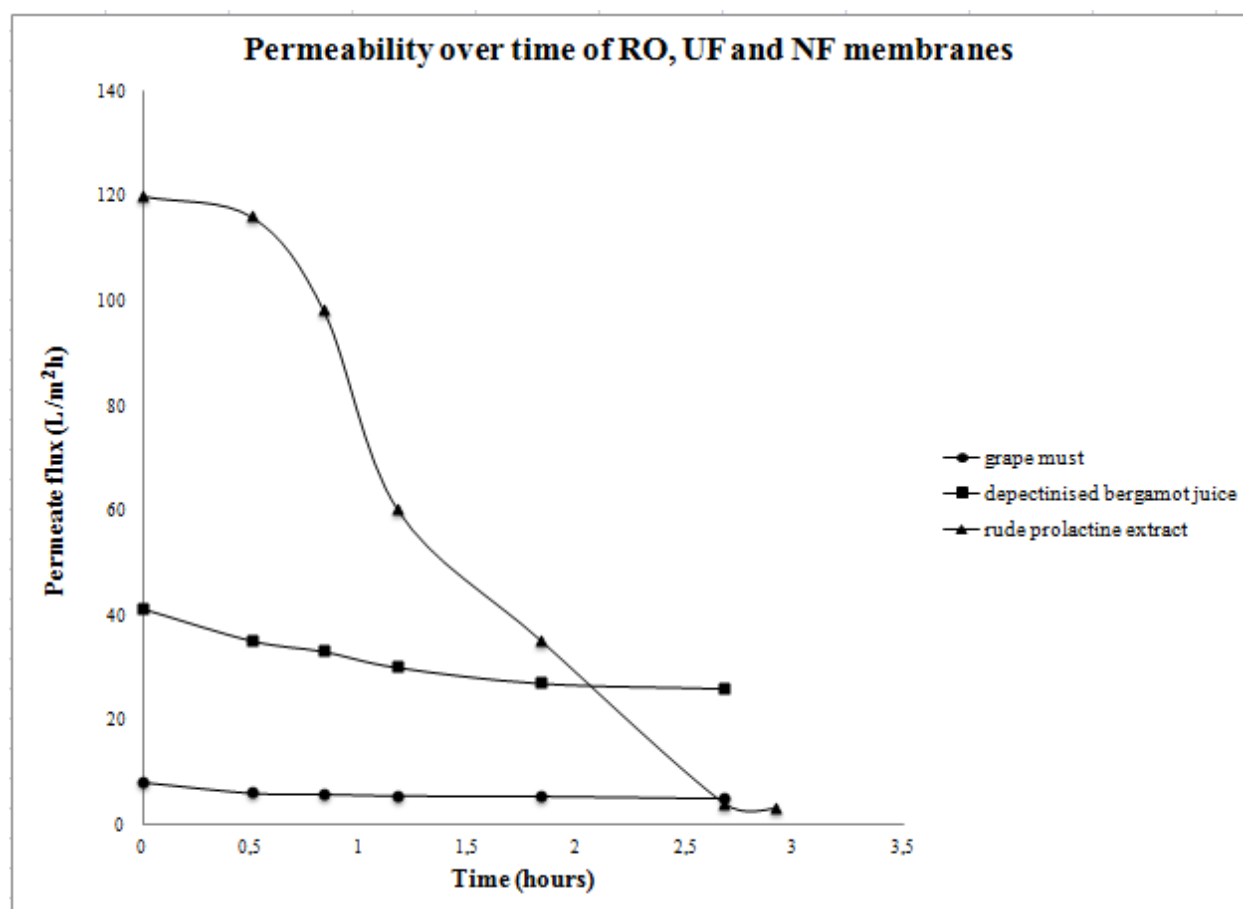


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|---|--|----------------------------------|-------------------------------------|-----------------------|
| Recovery of protein                             | Polysulfone membrane                                       | 25°C, pH:<br>6.74, TMP:<br>14psi | Poultry<br>processing<br>wastewater | Lo et al.,<br>2005    |
| Concentration of lactose<br>and calcium content | Koch TFC-SR3<br>polyamide thin-film<br>composite membranes | 30°C, TMP:<br>15 bar             | Dairies                             | Rice et al.,<br>2009a |

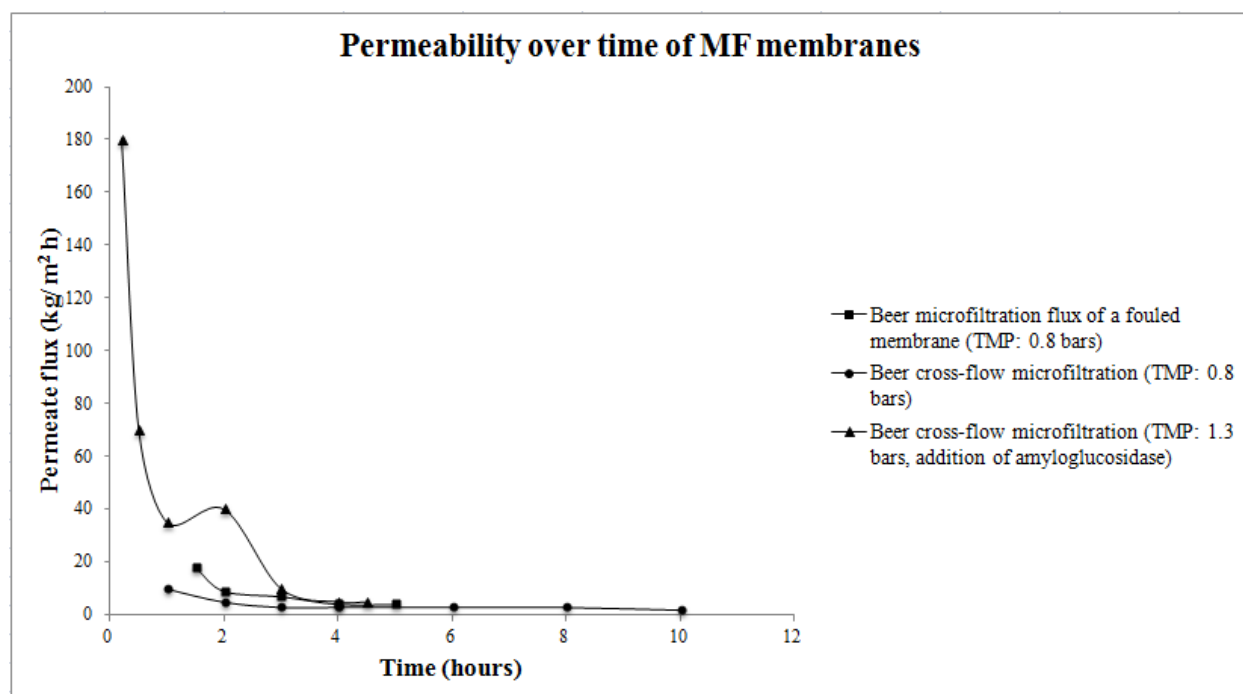
## 15. LEGENDS



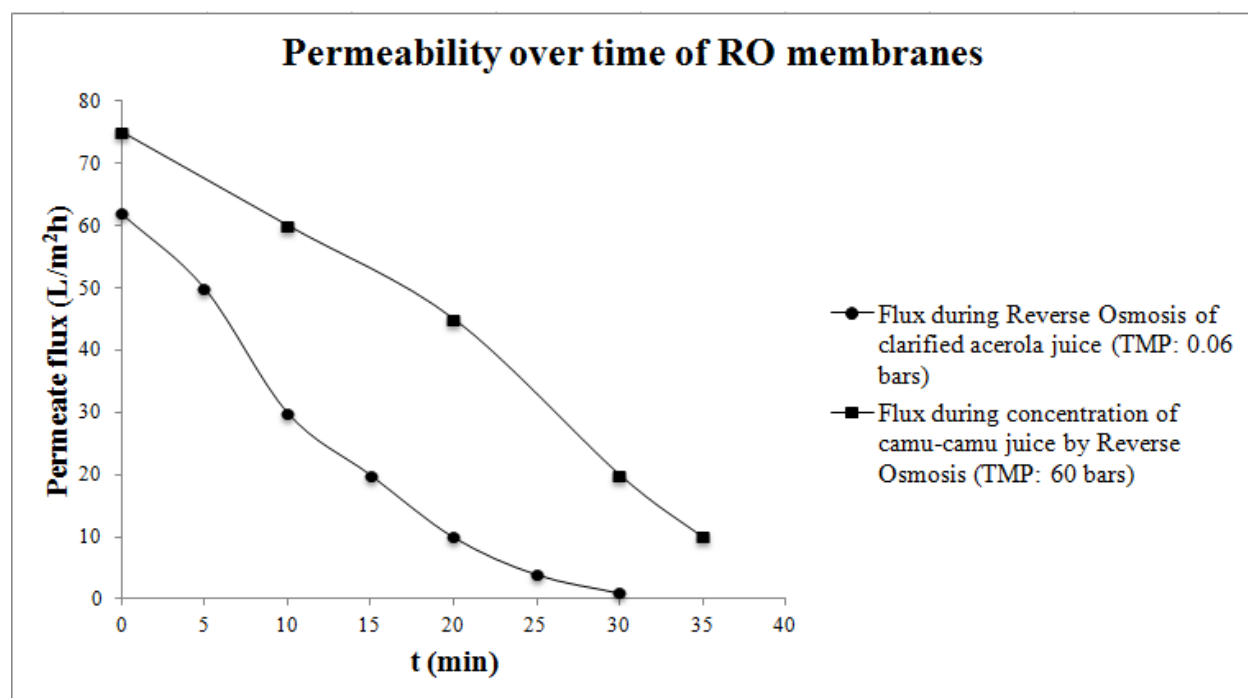
**Figure 1.** Permeate flux over time of MF membranes during the filtration of milk (TMP of 0.7 and 200 kPa) (Makardij et al., 1999; James et al., 2003).



**Figure 2.** Permeability over time of different membranes during the filtration of grape must (RO membrane, pressure: 55 bars, 17°C), depectinised bergamot juice (UF membrane, pressure: 7.5 bars, 24°C) and rude prolactine extract (tubular organic NF membrane, pressure: 35 bars, 55°C) (Ferrarini et al., 2001; Conidi et al., 2011; Bourseau et al., 2009).



**Figure 3.** Permeate flux over time during MF of beer using a fouled membrane (TMP of 0.8 bar, 3°C), MF of beer under TMP of 0.8 bar (2±0.7°C), and beer cross-flow MF with the addition of amyloglucosidase (TMP: 1.3 bars, <3°C) (Gan et al., 1999; Gan, 2001; Gan et al., 1997).



**Figure 4.** Permeate flux over time of RO membranes during the filtration of clarified acerola juice (TMP of 0.06 bars) and during concentration of camu-camu juice (TMP of 60 bars) (Matta et al., 2004; Rodrigues et al., 2004).