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# Fruit juice processing using membrane technology: A review

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ABSTRACT

Membrane technology has emerged as a substitute to traditional juice clarification and concentration processes as they require less manpower, reduce operating cost and low temperature. It is a low temperature process in which the organoleptic quality of the juice is almost retained. The advantages of these membrane processes over traditional methods are lower thermal damage to product, increase in aroma retention, less energy consumption, and lower equipment costs. Membrane concentration of fruit juice not only provides microbiological stability but also permits economy in packaging and distribution of the finished product due to a reduction in bulk by weight and volume. The biggest problem in the use of membrane based processes for the clarification/concentration of fruit juices is membrane fouling. Membrane fouling manifests itself as a decline in flux with the time of operation, reducing the membrane permeability. The degree of membrane fouling determines the frequency of cleaning, the lifetime of the membrane, the membrane area needed and consequently costs, design and operation of membrane plants. In this review, different membrane separation methods including microfiltration, ultrafiltration, nanofiltration and reverse osmosis for fruit juice clarification/concentration reported in the literature in the last fifteen years are discussed. Membrane Distillation methods for juice concentration is also covered in this review.

## 1. Introduction

Thermal processing remains the most widely employed method for shelf-life extension and food preservation and concentration. However, industrial thermal treatments may have negative impacts on nutritious components (such as anthocyanins, carotenoids, vitamins and bioactive proteins (Barros, Nunes, Gonçalves, Bennett, & Silva, 2011; Kechinski, Guimarães, Noreña, Tessaro, & Marczak, 2010; Provesi, Dias, & Amante, 2011; Van den Hout, Meerdink, & Vant Riet, 1999)) and sensory parameters (such as color, aroma, flavor (Nisha, Singhal, & Pandit, 2009; Timoumi, Mihoubi, & Zagrouba, 2007)). Membrane technology has emerged as the alternative to traditional thermal techniques for fruit juice clarification and concentration that were widely applied in the dairy and beverage industries. Membrane Separation methods are utilized in the food industry due to their less manpower requirement, greater efficiency and shorter processing time than conventional filtration. Consequently, the operational costs of using membrane processes are significantly lower than those of conventional processes (Nunes & Peinemann, 2001). Fruit juices are usually concentrated by multi-stage vacuum evaporation in order to reduce the storage and shipping costs, and to achieve stability and longer storage. However, loss of fresh juice flavors, color degradation and a "cooked" taste are some unwanted effects are associated with this method mainly due to the thermal impact. Researchers, over the years have tried to develop novel methods for retaining the flavor, aroma, appearance and mouth feel of freshly squeezed juices in the concentrate and ultimately in the reconstituted juice. Researchers have greatly succeeded in developing aroma retention, innovative process control and product blending methods to produce a good quality concentrate that can lead to consumer satisfaction, but not up to that level to make it readily unrecognizable from fresh juice. Significant efforts have been devoted in studying Ultrafiltration and Reverse Osmosis for juice clarification/concentration. Improved methods such as freeze concentration, sublimation concentration are also analyzed for juice processing (Koseoglu, Lawhon, & Lusas, 1990). But, based on recent research, the most encouraging alternative has to be membrane concentration. The types of pressure driven membrane separation processes which are most commonly used in juice processing are ultrafiltration (UF) and microfiltration (MF). They are able to separate particles in the approximate size ranges of 1–100 μm and 0.1–10 μm, respectively (Katasonova & Fedotov, 2009).

In recent times, advancements made in basic science and technology has enabled researchers to develop new membrane materials and improvements made in process engineering and intensification have helped overcoming major limitations of membrane based techniques. New membrane processes including membrane and osmotic distillation

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and integration of these techniques might contribute to the improvement of quality and make it economically feasible at an industrial level for fruit juice processing (Calabro, Jiao, & Drioli, 1994; Girard & Fukumoto, 2000). Wide variety of membrane modules including tubular, hollow fiber and spiral wound have been used in the food industry according to their advantages. They can be applied within the production process, i.e. for clarification and concentration, as well as for treating the resulting wastewater that is generated prior to disposal (sewer or surface discharge) or re-use.

Raw fruit juice contains lower molecular weight components like sugar, acid, salt, flavor, and aroma compounds. It additionally contains noteworthy measure of macromolecules (100–1000 ppm) for example, polysaccharides (pectins, cellulose, hemicellulose, and starch), dimness shaping segments (suspended solids (SSs), colloidal particles, proteins, and polyphenols) and so forth. Therefore in order to store for longer periods for commercial use, the juice needs to be clarified. Clarification is necessary for the removal of such macromolecules. In traditional processes, enzyme treatment of raw juice is being performed with the help of enzymes (pectinase and amylase) for reducing the pectic substances and starch content followed by addition of fining agents. This enzymatic treatment helps in reducing the cloudiness and viscosity and thereby makes the clarification process easier. The main function of the fining agents such as gelatin, bentonite etc. is to enhance the settling of formed flocs. Then suspended solids, colloidal particles, proteins etc. are removed by conventional filtration. To facilitate the filtration process, filter aids such as diatomaceous earth or kieselguhr are used. The above mentioned traditional methods to clarify fruit juice are batch processes and are highly labor-intensive and time-consuming. Also one major concern is the incomplete removal of additives (fining agents and filter aids) from product juice which can affect the taste of juice. (DasGupta & Sarkar, 2012)

Thermal evaporation is one of the most conventional techniques for fruit juice concentration. Despite its economic feasibility and technology, it does exhibit some disadvantages when applied to fruit juices. Even under vacuum, operating temperatures are still high enough to bring about significant deterioration in the product juice such as degradation of color, loss of nutritional characteristics, and the development of a “cooked” taste. For example, lipids and ascorbic acid can be oxidized, amino acids and sugars can undergo the Maillard browning reaction, and pigments, especially anthocyanin, carotenoids, and chlorophyll, can be degraded (Toribo & Lozano, 1986; Lozano & Ibarz, 1997; Mikkelsen & Poll, 2002; Kato et al., 2003; Maskan, 2006). Due to high temperatures of evaporation, loss of aroma compounds occur in fruit juices (Lin, Rouseff, Barros, & Naim, 2002; Ramteke, Eipeson, & Patwardhan, 1990; Nisperos-Carriedo & Shaw, 1990).

Hence, compared to these traditional methods, energy saving membrane operations such as microfiltration (MF) and ultrafiltration (UF), represent a valid alternative for the clarification of additive-free high-quality fruit juices with natural fresh taste (DasGupta & Sarkar, 2012). One of the important plus points of using membranes in the clarification/concentration of fruit juice is that traditionally used dead-end mode operated cartridge or bag filters, generates a lot of media waste that needs to be disposed of. But there is very little build up on the surface in crossflow membrane separation processes therefore media disposal problems are minimized. (Ionics Inc, 2004). This paper will review the recent significant progress on membrane processes for clarifying and concentrating fruit juices, including the use of microfiltration, ultrafiltration, reverse osmosis, direct osmosis concentration, membrane and osmotic distillation, and integrated membrane processes.

## 2. Membrane processes

Below we present different membrane processes commonly used in the food processing industry and their applications in fruit juice processing.

### 2.1. Microfiltration (MF)

In the fruit juice processing industry, the main purpose of MF is mainly clarification to remove suspended solids (SS), fat and high molecular weight (HMW) proteins. In dairy industry, MF is used to clarify cheese whey, as well as de-fat and reduce the microbial load of milk (Merin, 1986). Microfiltration can also be used to separate fruit juices into a fibrous concentrated pulp, and a clarified fraction free of spoilage micro-organisms. Fruit juice processing industry widely uses MF for juice clarification purpose (De Oliveira, Doce, & de Barros, 2012; Vaillant, Millan, Dornier, Decloux, & Reynes, 2001).

### 2.2. Ultrafiltration (UF)

UF is essentially utilized for fractionation, fixation and filtration. For instance, UF can be utilized to fractionate milk for cheese generation, i.e. the retentate part contains proteins, fat and certain insoluble and bound salts, while the permeate contains lactose and solvent salts (Brans, Schroën, van der Sman, & Boom, 2004). Concentration of skimmed milk using UF produces a high calcium and protein content product (Vyas & Tong, 2003) which is one of its major applications in the dairy industry.

The fruit juice industry also uses UF for both clarification and concentration depending upon the MWCO. For elucidation, the permeate instead of the retentate is the result of intrigue. UF is utilized to clear up a wide assortment of natural juices by evacuating polluting influences, for example, yeast, molds, microscopic organisms and colloids, together with proteins, tannins and polysaccharides, which all confers stability to the last item (Mohammad, Ng, Lim, & Ng, 2012).

### 2.3. Nanofiltration (NF)

NF is most normally used to isolate a mixture that has a blend of some attractive parts and some that are not alluring. A case of this is the concentration of lactose syrup (Zhang, Yang, Zhang, Zhao, & Hua, 2011). It facilitates the passing of water through the membrane and at the same time holding back the sugar, and thereby concentrating the solution. NF is also effective in concentrating divalent salts, bacteria, proteins and other constituents that have a molecular weight $> 1$ kDa.

NF can be utilized to in part demineralization, and in addition to concentration. NF is a moderately new process for the demineralization of whey (Pan, Song, Wang, & Cao, 2011) in the dairy industry. In the juice processing industry, NF can be used to concentrate useful bioactive compounds from fruit juices e.g. lycopene in case of watermelon juice (Arriola et al., 2014).

### 2.4. Reverse osmosis (RO)

The main application of RO in food processing industries is to concentrate, purify and recover valuable components. RO can also be used in combination with other membrane separation processes, such as MF and UF. RO requires less operational cost due to evaporation, or even the elimination of this step (Hedrick, 1983; Merson, Paredes, & Hosaka, 1980). The energy requirements of RO have been shown to be significantly less than for mechanical vapor compression. RO is also 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 (Kotsanopoulos & Arvanitoyannis, 2015). RO's other advantages include:

- Quality of separation
- Minimal heat damage
- Low amount of waste generation and treatment
- Smaller footprint
- Lower capital requirements

Some disadvantages include restricted working pressure territory specific to some applications and film fouling with certain feed stocks.

### 2.5. Electrodialysis (ED)

Electrodialysis is a comparatively new process in terms of its use in food industry. Some applications of ED in food industry are as follows:

- Demineralization of milk and whey (Andres, Riera, & Alvarez, 1995)
- De-acidification of fruit juice (Vera, Ruales, Dornier, Sandeaux, Persin, et al., 2003; Vera, Ruales, Dornier, Sandeaux, Sandeaux, et al., 2003)
- De-ash sugar solutions (Boye & Arcand, 2012)

### 2.6. Pervaporation

Pervaporation is defined as a separation technique in which a liquid feed mixture is separated by partial vaporization through a non porous permselective membrane. Because pervaporation is based on a solution diffusion mechanism, it can be used to solve separation problems encountered with traditional, equilibrium-based, separation techniques (Karlsson & Tragardh, 1996). For the food industry, the following applications are being researched:

1) Alcohol removal from wine (Takács, Vatai, & Korány, 2007)
2) Aroma recovery from fruit juices, beer, herbal and flowery extracts (Catarino, Ferreira, & Mendes, 2009; Pereira et al., 2005)
3) Restoration of aroma components during fermentation (Schafer, Bengtsem, Pingel, Bödeleker, & Crespo, 1999)

Pervaporation is, however, despite its successes and potentials, so far not established in the food industry.

### 2.7. Membrane contactors – osmotic distillation

Membrane contactors achieve a gas liquid or liquid/liquid mass transfer of one phase to another without dispersion by passing phases on both sides of a microporous membrane. Controlling the pressure difference between the two phases carefully, one of the phases can be immobilized in the pores of the membranes and an interface between the two phases can be established at the mouth of each pore (Lipnizki, 2010). The concentration, and/or pressure difference between the feed and the permeate side is the driving force. Some of its applications in the food industry are:

1) Soft drinks carbonation (Klaassen, Feron, & Jansen, 2005)
2) Alcohol removal by osmotic distillation (Varavuth, Jiraratananon, & Atcharyawut, 2009)
3) fruit juice concentration (Cassano, Conidi, Timpone, D'avella, & Drioli, 2007; Cassano, Donato, & Drioli, 2007; Cassano, Marchio, & Drioli, 2007).

## 3. Membrane fouling and concentration polarization in juice processing

One of the main problems of using of membranes for clarifying or concentrating fruit juices is membrane fouling and concentration polarization. Concentration polarization is reversible fouling while pore blocking is mostly irreversible. Concentration polarization and pore blocking results in flux decay with the process time, thus reducing the permeability of the membrane or making a thin layer over the membrane surface. Membrane fouling controls the frequency of cleaning, the lifetime of the membrane, area needed for separation which ultimately determines the costs, design and operating parameters of membrane plants. Membrane fouling is a complicated phenomenon due to the involvement of various colloidal particles present in the juice in making the cake layer over the membrane surface or blocking the membrane pores. The fouling materials in fruit juice are mainly composed of cell wall polysaccharides and macromolecules, such as pectins (polyuronic acids mostly derived from -galacturonic acid, primarily plentiful in apple and citrus fruits), cellulose, lignin and hemicelluloses. The main problems generated by build-up of these foulants are the reduction in flux and changes of physico-chemical properties of filtrate. Therefore a major portion of research is centered around at effectively mitigating membrane fouling and significant restoration of membrane flux. Nevertheless, the complete elimination of the fouling is impossible (Madaeni, Mohamamdi, & Moghadam, 2001)

### 3.1. Methods of reducing concentration polarization and membrane fouling

Pretreatment methods before juice clarification can reduce the particulate suspended materials in the juice, resulting in improvement of flux and attainment of higher concentration factors. The first step for pretreatment for clarification of citrus juices using membrane separation is pectin removal with the help of enzymes.

The most used method for pretreatment of juices to remove pectin is enzymatic treatment by pectinase. To reduce the viscosity of the juice, mixtures of enzymes, known as pectinases, are used to hydrolyse pectin into poly-d-galacturonic acid fragments, with relatively little pulp which essentially leads to an increase in permeate fluxes and yield recovery (Alvarez, Alvarez, Riera, & Coca, 1998). More than 95% recovery of the product can be achieved by UF of such depectinised juices. Combined effect of pectinase and cellulase was successful in enhancing permeate flux in microfiltration of passion fruit with ceramic membranes (Vaillant, Millan, Brien, & Decloux, 1999). They found minimum viscosity and high clarity to be the optimum conditions for depectinisation.

Proteins, fibers, suspended solids etc. are some other components which affect the filtration efficiency of juice. Therefore, in addition to depectinisation, various other pretreatments can also be used to improve the performance juice clarification or concentration. They include treatment with proteases for the removal of proteins (Pinelo, Zeuner, & Meyer, 2010), centrifugation (before or after depectinization) (Yousefnezhad, Mirsaeedghazi, & Arabhosseini, 2016; Rai, Majumdar, Dasgupta, & De, 2007) and use of fining agents, such as gelatine and bentonite (Youn, Hong, Bae, Kim, & Kim, 2004).

Rai et al. (2007) investigated the impact of some pre-treatment methods to determine the efficiency of UF for clarification of mosambi juice. The pretreatment methods include centrifugation, fining with gelatin and with both bentonite and gelatin, pectinase treatment, centrifugation after enzymatic treatment and enzymatic treatment succeeded by fining with bentonite. After analysis all these methods they concluded that pectinase treatment succeeded by fining with bentonite was most successful in enhancing permeate flux for the same pressure. They observed a 77% increase in flux as compared with the only enzymatic treatment.

A few other flux enhancing techniques have additionally been explored with a specific end goal to diminish fouling layer and pore blocking in juice processing. They include ultrasonic vibration, periodic backwashing with air or $N_2$ (Su, Liu, & Wiley, 1993) and backwashing by pulsating flow (Ben Amar, Gupta, & Jaffrin, 1990), use of turbulence promoters (Pal et al., 2008) etc. High pulp content and high viscosity of the juice limits the performance of UF. Zhu, Mhemdi, Ding, et al. (2015) found out that for high pulp content, vibrating and rotating membrane systems are more appropriate due to the amount of shear rate created at the membrane interface leading to optimum control of fouling phenomena.

Reducing the interaction between proteins and other foulants with the membrane using membrane surface modifications can be also helpful in reducing fouling phenomena (Rana & Matsuura, 2010). A narrow pore size distribution can reduce the fouling compared to more porous membranes such as MF membranes. Hydrophobic membranes are more prone to fouling compared to hydrophilic membranes (Kumar & Ismail, 2015).

## 4. Membrane modules

Different configurations of membrane modules have been used by researchers to examine the effect on fruit juice clarification and concentration. The most commonly used modules are plate-and-frame (He, Zhijuan, & Shunxin, 2007), hollow fiber (Laorko, Li, Tongchitpakdee, & Youravong, 2011) tubular (Cassano et al., 2007), spiral wound (Ghosh, Balakrishnan, Dua, & Bhagat, 2000), and dead-end or stirred batch cells (Riedl, Girard, & Lencki, 1998). In the plate-frame module, two flat sheet membranes are sandwiched around a support plate. The membranes are attached to the plate with the help of either a gasket with locking devices, glue or directly bonded. The internally porous plate provides a flow channel for permeate which is collected from a tube on the side of the plate. The feed channel can be a clear path with channel heights from 0.3 to 0.75 mm. Tubular membranes primarily function in a tangential or cross-flow mode. Here, the feed solution is pumped at high velocities along the center of the tube. High cross-flow feed velocities helps in mitigating the development of a concentration polarization layer over the membrane surface leading to high and stable flux and easy cleaning. Tubular modules are superior compared to other modules w.r.t. their ability to process solutions containing high suspended solid, and for achieving higher concentration levels without plugging. For this reason they are widely utilized for juice concentration in juice processing industry. In spiral wound membrane, membranes are casted as a film onto flat sheets and are sandwiched together with feed spacers (typical thickness 0.03 to 0.1 in) and permeate carrier. They are sealed at each edge and wound up around a perforated tube. The thin spacers help in establishing the desired feed channel height. The open end of the envelope is sealed around the perforated tube. The feed is fed at one end of the module and flows along the length of the module while the retentate is collected from the end of the tube. The permeate flows through the membrane into the permeate channel and spirals toward the perforated center tube. The comparison of important characteristics of different membrane modules are listed in Table 1.

## 5. Clarification & concentration of fruit juices using membrane process

### 5.1. Effect of operating parameters in permeate flux

Permeate flux decline with time due to concentration polarization and fouling remains a major hindrance in fruit juice processing using membrane methods. Researchers have tried to maintain high flux levels by optimizing different operating conditions to get maximum efficiency. He et al. (2007) optimized different parameters for ultrafiltration of apple juice and listed TMP = 2.0 bar, CFV = 2.5 m/s and $T$ = 50 °C as best suited conditions for the study. De Bruijn, Venegas, Martiınez, and Borquez (2003) compared flux and energy usage at different pressure and velocities for ultrafiltration of apple juice but couldn't find a unique optimum operating condition. Most researchers have found that an increase in temperature has a positive effect in flux enhancement in fruit juice clarification processes (Vladisavljevic, Vukosavljevic, & Bukvic, 2003; Cassano, Donato et al., 2007; De Barros, Andrade, Mendes, & Peres, 2003; Tasselli, Cassano, & Drioli, 2007; Cassano, Mecchia, & Drioli, 2008). Youn et al. (2004) performed MF of apple juice at 25 °C under the operation pressure of 1.5 kgf/cm$^2$ and flow rate of 200 mL/min, where the reduction of permeate flux was < 20% even after 60 min. Pagliero, Ochoa, and Marchese (2011) found a maximum value of clarified permeate flux of 47 L/m$^2$ h at P = 1 bar and $v$ = 1.25 m/s at a temperature of 25 °C in orange juice clarification. An increase in flow rate also helps in increasing flux (Cassano, Donato et al., 2007; Cassano, Marchio et al., 2007). High values of pressure helped in maintaining a high level of flux during reverse osmosis concentration of fruit juices (Jesus et al., 2007). A trend of declining permeate flux is generally observed with an increase of volumetric concentration factor in case of fruit juice concentration processes (Cassano, Donato et al., 2007; Cassano, Marchio et al., 2007; Jesus et al., 2007). A high operating pressure of 7.5 bar resulted in a higher sugar recovery in ultrafiltration of pineapple juice (Carvalho, Castro, & Silva, 2008). Laorko, Li, Tongchitpakdee, and Youravong (2010) found a linear increase in flux with increase in crossflow velocity (Fig. 1) in pineapple juice clarification due to the enhance wall shear stress on the membrane surface. Similar trend was found by Gomes et al. (2013) in microfiltration of watermelon juice and Hojjatpanah et al. (2011), Sarkar, DasGupta, and De (2009), Cassano et al. (2015) and Ushikubo, Watanabe, and Viotto (2007) for the clarification of black mulberry, mosambi, pomegranate and umbu juice respectively. The authors attributed this increase in permeation flux to the removal of surface solute particles due to the use of a higher cross-flow velocity, which lead to a reduction in fouling. Increase in temperature also had a significant flux enhancement effect in osmotic evaporation of pineapple juice (Hongvaleerat, Cabral, Dornier, Reynes, & Ningsanond, 2008). The author pointed this increase of flux with temperature to an increase of water partial pressure at the liquid–gas interface at the juice side of the membrane, which increases the driving force for water transfer. Researchers found an increase in steady state permeate flux with an increase in feed flow rate in fruit juice clarification processes (Tasselli et al., 2007; Cassano, Donato et al., 2007). Tasselli et al. (2007) and Cassano, Donato, et al. (2007) found 75 kPa and 90 kPa as optimum operating pressures in kiwifruit juice ultrafiltration using PEEK and PVDF membranes. Considering maximum permeation flux, minimum fouling and quality of kiwi fruit juice as the requirements, Cassano, Dopnato, et al. (2007) identified the best conditions to be at 25 °C of temperature, 90 kPa of pressure and 700 l/h of flow rate. Nourbakhsh et al. (2014) studied the microfiltration of watermelon juice in a cross-flow membrane system and demonstrated that the total resistance to permeation flux decreased by approximately 54% when feed temperature was increased from 20 to 50 °C. Sarkar et al. (2009) found an increase of permeate flux with an increase in electric field (voltage/metre) in cross-flow electro ultrafiltration of mosambi juice. Researchers have noticed the rapid decline of flux in the initial periods of fruit juice clarification/concentration (Cassano, Donato et al., 2007; Cassano, Marchio et al., 2007; Cassano et al., 2008; Rezzadori, Serpa, Penha, Petrus, & Petrus, 2014; Ghosh et al., 2000; Baklouti, Ellouze-Ghorbel, Mokni, & Chaabouni, 2011; Mirsaeedghazi, Emam-Djomeh, Mousavi, Ahmadkhaniha, & Shafiee, 2010; Mirsaeedghazi, Emam-Djomeh, Mousavi, Aroujalian, & Navidbakhsh, 2010 etc). They have linked this to the deposition and growth of a polarized layer formed by high molecular weight compounds present in fruit juices. Fig. 2 shows

**Table 1**
Comparison of different membrane modules.

| Module | Packing density (m$^2$/m$^3$) | Energy cost (pumping) | Channel spacing (cm) | Particulate plugging | Ease of cleaning | Hold up volume |
|---|---|---|---|---|---|---|
| Flat plate | 300 | Moderate | 0.03–0.25 | Moderate | Good | Low |
| Tubular | 60 | High | 1–2.5 | Low | Excellent | High |
| Hollow fiber | 1200 | Low | 0.02–0.25 | High | Fair | Low |
| Spiral wound | 600 | Low | | Very high | Moderate | Low |

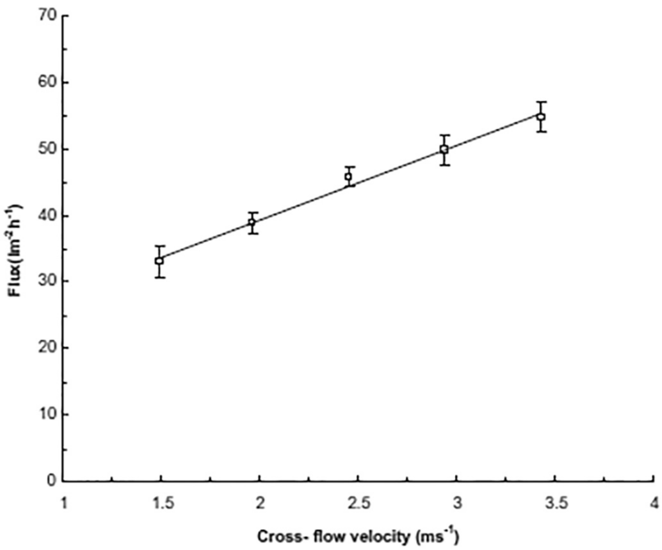


**Fig. 1.** Effect of cross-flow velocity on the permeate flux (membrane pore size 0.2 μm, TMP = 0.7 bar, temperature = 20 ± 2 °C) (Laorko et al., 2010).

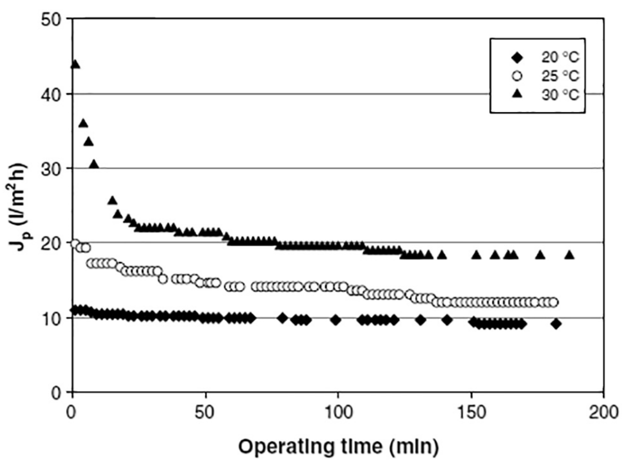


**Fig. 2.** Time course of permeate flux at different temperatures (operating conditions: TMP = 115 kPa; $Q_f$ = 500 l/h) (Cassano, Donato et al., 2007).

the flux decline trend with time in ultrafiltration of kiwifruit juice (Cassano, Donato et al., 2007).

### 5.2. Effect of pretreatment in permeate flux and juice quality

The importance of feed pretreatment in augmentation of permeates flux and overall process efficiency has been covered in the Membrane Fouling section. Over the years, researchers have tried various methods of pretreatment to enhance fruit juice clarification/concentration processes. Enzymatic treatment has been widely used as a pretreatment in juice processing industries (Aguiar et al., 2012; Yazdanshenas, Tabatabaeenezhad, Roostaazad, & Khoshfetrat, 2005; Toker, Karhan, Tetik, Turhan, & Oziyci, 2013; Carvalho et al., 2008; Laorko et al., 2011; Vaillant et al., 2005; Rai, Majumdar, Sharma, Das Gupta, & De, 2006; Baklouti et al., 2011). Researchers have shown that Enzymatic pretreatment helps in maintaining a higher flux level in case of clarification/concentration of citrus juices by reducing the juice viscosity and pectic materials (AIS) (He et al., 2007; Maktouf et al., 2014; Nandi, Uppaluri, & Purkait, 2009; Youn et al., 2004). Fig. 3. shows high levels of flux during apple juice pretreated by enzymatic treatment and pasteurization and clarified by using UF (He et al., 2007). Gelatin and Bentonite have also been used in pre-clarification of juices to ease the load in subsequent membrane filtration processes (Toker et al., 2013;

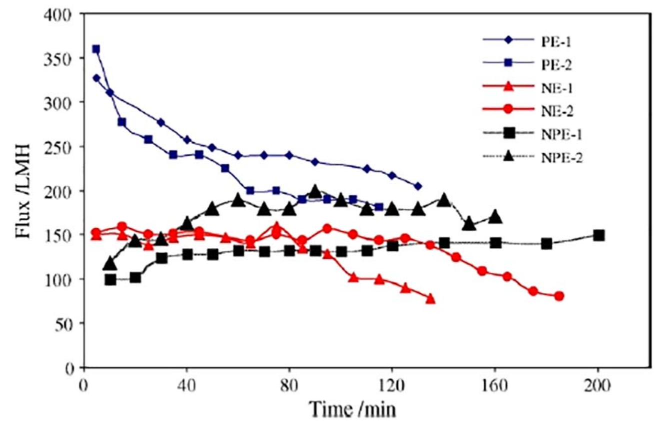


**Fig. 3.** The flux vs. time during clarifying apple juice differently pretreated. TMP = 2.0 bar, CFV = 2.5 m/s (He et al., 2007).

Onsekizoglu, Bahceci, & Acar, 2010). These fining agents retain the suspended solids responsible for the fouling of the membrane such as phenolic substances and proteins, in feed as large aggregates by the virtue of their electrostatic and adsorptive effects. Pectolytic enzymes are used successfully to degrade pectin which is responsible for forming a gel layer over the membrane surface (Alvarez et al., 1998; Vladisavljevic et al., 2003; Onsekizoglu et al., 2010; Carvalho et al., 2008; Laorko et al., 2010; Cassano, Donato et al., 2007; Maktouf et al., 2014; Nandi et al., 2009; Sarkar et al., 2009; De Oliveira et al., 2012; Ushikubo et al., 2007). Depectinisation also helps in reducing the juice viscosity which ultimately enhances permeate flux. Fig. 4 shows effect of enzymatic treatment on apple juice viscosity (Alvarez et al., 1998). De Bruijn et al. (2003) found that enzymatic preparations were successful in hydrolysing polysaccharides such as pectin, starch, cellulose and hemicellulose prior to UF, thereby improving membrane performance in apple juice clarification. Centrifugation as a pretreatment has also been used to remove enzyme molecules, large haze particles and adjust the pulp content (Vladisavljevic et al., 2003; Saura et al., 2012; De Oliviera et al., 2012). Pretreatment using filter aids such as bentonite has a positive effect on enhancing flux of subsequent membrane filtrations for juice clarification (Youn et al., 2004). This can be attributed to their ability to remove haze compounds by adsorption. Preclarification with chitosan has been successful in reducing turbidity and viscosity before fruit juice concentration (Domingues, Ramos, Cardoso, & Reis, 2014). Enzymatic pretreatment does not alter the total soluble solids content in the juices significantly but improves the clarity of the juice (Domingues et al., 2014; Nandi et al., 2009). Pulsed electric

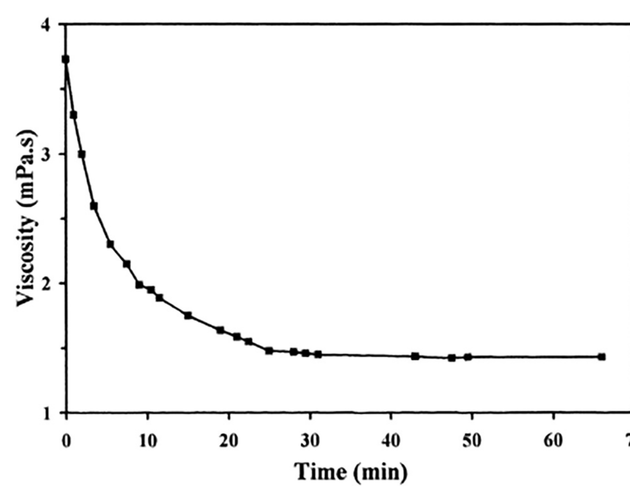


**Fig. 4.** Influence of enzymatic treatment with Pectinex 3XL on apple juice viscosity (Alvarez et al., 1998)

field has also been used as a pretreatment in chicory juice clarification with satisfactory results (Zhu et al., 2013).

### 5.3. Effect of membrane treatment on juice quality

Microfiltration and Ultrafiltration have been widely used for fruit juice clarification in recent years. Ultrafiltration is also used for concentration based on its molecular weight cut-off. Ultrafiltration with a 50 kDa PES membrane produced a good quality clarified apple juice with acceptable color, clarity and turbidity values with the rejection of pectin, starch and thermo- acidophilic bacteria (He et al., 2007). Similar commercial specifications in color, clarity and turbidity were achieved by De Bruijn et al. (2003) and Vladisavljevic et al. (2003) using inorganic membranes for apple juice clarification. They also found that the larger MWCO membrane had a positive effect on shelf life of the juice. With regard to soluble solids and acids in the clarified apple juice, UF treatments didn't show any significant difference (Zarate-Rodriguez et al. 2001; Youn et al., 2004; Vladisavljevic et al., 2003; De Bruijn et al., 2003). Valdisavljevic et al. (2003) analyzed that polyphenolic content in the apple juice permeate decrease with the decrease in MWCO of the UF membranes. Zarate-Rodriguez et al. (2001) observed browning in clarified apple juice with a higher MWCO membrane due to the development of polyphenols-oxidase (PPO) during storage. Research of Youn et al. (2004) showed that Ultrafiltration with a 30 kDa membrane didn't have a notable change of vitamin C in the clarified apple juice. Warczok, Ferrando, Lopez, and Guell (2004) used nanofiltration for concentration of apple juice and achieved a good degree of fructose concentration. Reverse Osmosis produced a 3 fold increase in soluble solids content of concentrated apple juice and with further concentration with osmotic evaporation, the value rose to 6 fold. (Aguiar et al., 2012), though in both cases, total phenolics and antioxidant capacity loss was significant. Comparison of different physicochemical properties analyzed by Aguiar et al. (2012) is listed in Table 2. Onsekizoglu et al. (2010) used ultrafiltration followed by osmotic distillation, membrane distillation or a combination of these methods, in order to concentrate apple juice up to 65 °Brix. They found similar nutritional and sensorial characteristics of the concentrated juice compared to that of the original juice. They were particularly successful in retaining bright natural color and pleasant aroma in the concentrate that were lost during thermal evaporation. The authors concluded that clarification using ultrafiltration membrane led to a reduction of total polyphenolic content, however the concentration processes did not significantly affect this property.

Microfiltration and Ultrafiltration of orange juice produces a clear juice removing the suspended solids and retaining almost the same amount of soluble solids and acids (Pagliero et al., 2011; Toker et al., 2013). Similar result was found in case of UF also by Cassano, Marchio, et al. (2007) with the addition that polyphenolics were well pre served in the clarified orange juice. Toker et al. (2013) found that ascorbic acid and phenolic content in the clarified orange juice was higher for higher MWCO membrane. A higher value of total antioxidant activity (TAA) in the product is very much desirable due to their role in reducing the risk of free radical related oxidative damage associated with a number of diseases. Galaverna et al. (2008) evaluated changes in some important physicochemical properties during integrated membrane processing of blood orange juice. They found nearly same amount of antioxidant compounds apart from anthocyanins and Vitamin C in the concentrate as that of the feed juice during the process, though there is about 15–20% decrease in the contents of anthocyanins and Vitamin C in the concentrate. Only 15% reduction of TAA was found in the blood orange juice concentrate. The concentrate retains its bright red color and its pleasant flavor. Reverse Osmosis produced concentrated orange juice with a high percentage of soluble solids and vitamin c which showed a increasing trend with increasing pressure (Jesus et al., 2007). Reverse Osmosis concentrate also preserved the characteristic aroma of the juice, differing significantly from the juice concentrated by thermal evaporation. Their results are shown in Table 3. Mirsaeedghazi and Emam-Djomeh (2017) was successful in clarifying bitter orange juice using microfiltration with almost 98% turbidity removal and acceptable levels of polyphenols content in the final product.

The use of membrane technology for the concentration of lycopene, a carotenoid with high antioxidant capacity from watermelon juice is feasible and represents a good alternative for the industrial production of this compound. The molar mass of lycopene is 536.85 Da and is therefore retained on the concentrate side of the membrane. Rai et al. (2010) used microfiltration and achieved an approximately threefold increase in the lycopene content of the retained stream of watermelon juice. Gomes et al. (2013) attained an increase of 400% under optimum operating conditions during MF of watermelon juice. Soluble solids content remain almost the same in the clarified watermelon juice during MF (Chaya et al. 2008). Nanofiltration has also been able to achieve a great level of lycopene concentration during watermelon juice processing (Arriola et al., 2014). Vaillant et al. (2005) performed osmotic evaporation of melon juice and observed a loss of about 30% of phenolic compounds. They associated this loss with polyphenol oxidases present in the feed juice, which acted during processing. Bhattacharjee, Saxena, and Dutta (2017) analyzed the effect of ultrafiltration of watermelon juice on its sugar and vitamin C content. For increase in UF concentration factor, they found an increasing trend for sugar but vitamin c content decreased. The authors compared concentration factors of 1.6 and 2.5 and concluded that 1.6 will be more effective with regard to both sugar and vitamin C concentrations.

The retention of sugar in the process of clarified pineapple juice by MF and UF was studied by Carvalho et al. (2008). The researchers

**Table 2**
Physicochemical characteristics of concentrated apple juice obtained by reverse osmosis and osmotic evaporation (Aguiar et al., 2012).

| Analysis | Feed | Reverse osmosis | Osmotic evaporation |
|---|---|---|---|
| pH | 3.83[a] | 3.79[b] | 3.67[c] |
| Soluble solids (°Brix) | 8.7[c] | 28.1[b] | 51.2[a] |
| Total solids (g/kg) | 92.2[c] | 291.1[b] | 526.8[a] |
| Titratable acidity (g/kg) | 20.2[c] | 64.4[b] | 119.0[a] |
| (g/kg dry matter) | 219.1[a] | 221.2[a] | 225.9[a] |
| Total phenolics (mg GA/kg) | 495.3[c] | 1393.7[b] | 2328.4[a] |
| (mg GA/kg dry matter) | 5372.0[a] | 4787.7[b] | 4419.9[c] |
| Antioxidant activity (mmol TE/g) | 5.9[c] | 13.2[b] | 18.6[a] |
| (mmol TE/g dry matter) | 57.4[a] | 45.3[b] | 35.4[c] |

Different letters indicate significant difference between different samples ($p \le 0.05$), determined by Tukey's test.

**Table 3**
Chemical and physical evaluation of single strength orange juice (feed) and concentrated juices obtained at different transmembrane pressures (Jesus et al., 2007).

| | Single strength juice | Concentrated juice | | |
|---|---|---|---|---|
| | Feed | 20 bar | 40 bar | 60 bar |
| pH | 4.2 | 4.1 | 4.3 | 4.3 |
| Acidity (g de citric acid/100 ml) | 0.4 | 1.0 | 1.4 | 1.8 |
| Soluble solids (°Brix) | 8.2 | 16.0 | 28.5 | 35.7 |
| Pulp content (%g/g) | 3.4 | 4.9 | 22.5 | 28.7 |
| Viscosity (mPa s) | 1.5 | 2.3 | 5.3 | 10.3 |
| Vitamin C (mg de ascorbic acid/100 g) | 29.3 | 53.9 | 82.7 | 101.1 |
| Luminosity | 14.4 | 11.0 | 7.9 | 7.7 |
| $a_{Hunter}$ | 1.0 | 3.0 | 5.2 | 5.8 |
| $b_{Hunter}$ | 9.9 | 7.0 | 4.4 | 4.3 |

L, luminosity (0 = black and 100 = white); a (− 80 to zero = green, from zero to + 100 = red); b (− 100 to zero = blue, from zero to + 70 = yellow).

found sugar content was influenced by the membrane pore size and MWCO as well as the geometry of the module. Laorko et al. (2010) performed both MF and UF of pineapple juice and found MF more suitable with the highest recovery of phytochemical compounds including vitamin C (94.3%), total phenolic content (93.4%) and DPPH free radical scavenging capacity (99.6%). UF didn't affect the total soluble solids content of pineapple juice (De Barros et al., 2003). Osmotic evaporation gave satisfactory levels of concentration with regard to soluble solids and phenolics for pineapple juice (Hongvaleerat et al., 2008).

Mirsaeedghazi, Emam-Djomeh, Mousavi, Aroujalian et al. (2010) achieved a good level of clarification of pomegranate juice using Microfiltration. Suspended solids were completely removed reducing turbidity while retaining nearly all soluble solids. Similar results were found by Cassano, Conidi, and Tasselli (2015) using UF for pomegranate juice clarification. The findings of Cassano et al. (2015) is listed in Table 4. Comparing MF and UF for pomegranate juice clarification, Mirsaeedghazi et al. (2012) concluded that MF is better with flux and fouling considerations as clarified juices from both process having similar physicochemical properties. Significant decrease in phenolic content of pomegranate juice following microfiltration was reported by Mirsaeedghazi, Emam-Djomeh, Mousavi, Ahmadkhaniha et al. (2010), however the ratio of reduction was not reported. Individual acids such as citric acid, malic acid and quinic acid were fully retained while a decrease in antioxidant capacity in clarified pomegranate juice was observed by Bagci (2014) after UF. The author also noticed a significant reduction of total anthocyanin content following UF of raw pomegranate juice. Conidi, Cassano, Caiazzo, and Drioli (2017) investigated concentration of phenolic compounds in pomegranate juice using ultrafiltration and nanofiltration membrane. They were successful in retaining a high percentage (85%) of polyphenols in the retentate portion. They performed diafiltration for recovering a high percentage of sugar in permeate and diafiltrate fraction which can be reused as food additives or as bases for soft drinks.

Microfiltration using ceramic membranes improved color and clarity of mosambi juice significantly (Nandi et al., 2009). However, the valuable properties of the juice such as pH, soluble solids, acidity, density does not vary significantly for the process. Those results match with the outcome of Rai et al. (2006), who used polymeric membrane for mosambi juice clarification. Electro-ultrafiltration also achieved a great degree of clarification of mosambi juice showing a trend of increasing clarity with the increase in electric potential (Sarkar et al., 2009). The author also observed a satisfactory level of soluble solids concentration in the clarified juice during the process.

Vaillant Jeanton, et al. (2001) evaluated the potential of osmotic evaporation for concentrating clarified passion fruit juice on an industrial scale at 30 °C, up to total soluble solids (TSS) higher than 60 °Brix. They used a pilot plant containing a 10 $m^2$ hollow-fiber module. At 40° and 60 °Brix, average evaporation fluxes of 0.65 kg/$m^2$/h and of 0.50 kg/$m^2$/h were obtained respectively. These concentrate values were 10 times lower than those obtained in RO for the same value of flux. Sensory quality and vitamin C content were well preserved in the concentrated juice. Shaw et al. (2001) evaluated the retention of flavors in concentrated orange and passion fruit juices (previously clarified by MF) obtained by using a pilot-scale osmotic evaporator containing 10.3 $m^2$ of PP hollow fibers. Both juices were concentrated threefold to 33.5 and 43.5 °Brix, respectively. Their results also showed a loss of volatile compounds of about 32% and 39% in orange and passion fruit juice, respectively. Microfiltration of passion fruit juice process was able to reduce color and turbidity of the feed juice, resulting in a visually clean product (Domingues et al., 2014). Similar results were found by De Oliviera et al. (2012) by using MF for passion fruit clarification.

Grape juice concentration up to 28.5 °Brix was attained by using reverse osmosis process (Gurak, Cabral, Rocha-Leao, Matta, & Freitas, 2010). The concentrated grape juice presented an increase in total titrable acidity, anthocyanin and phenolic compound contents, color density and color index proportional to the volumetric concentration factor. Cassano et al. (2008) achieved a high degree of clarity by using UF for grape must processing. TSS and phenolic content also was satisfactory whose values decreased with increasing pressure as shown in Table 5.

The application of UF to lemon juice caused notable reductions in turbidity (99%) and viscosity (98%), subsequently achieving a high level of clarity (Maktouf et al., 2014). To use as an acidifier or flavor additive in the food industry, Saura et al. (2012) used MF and UF to clarify lemon juice. They studied different volatile fractions and found selective rejection of terpene hydrocarbons, the most abundant fraction. This de-terpenation process is of vital importance because of the negative characteristics of the terpene hydrocarbons for the aromatization of food items, e.g. they are not soluble in water and accumulate in small drops on the surface of the beverages or preservation liquid. Clarified permeate lemon juice by microfiltration presented titrable acidity, pH and TSS values comparable with those of untreated fresh lemon juice (Espamer, Pagliero, Ochoa, & Marchese, 2006). Similar results were found by Chornomaz, Pagliero, Marchese, and Ochoa (2013).

Ultrafiltration with a polyether ether ketone membrane was able to produce a clarified kiwifruit juice free of suspended solids with a small amount of 16% loss in soluble solids (Tasselli et al., 2007). The clarified juice had a large improvement in color and clarity. Cassano, Donato, et al. (2007) achieved a clarified kiwifruit juice with a 11% loss in TSS and 16% loss in ascorbic acid using ultrafiltration with a polyvinyledeneflouride membrane as the clarifying method. Bánvölgyi, Horváth, Stefanovits-Bányai, Békássy-Molnár, and Vatai (2009) evaluated the effect of concentration by reverse osmosis on the characteristics of blackcurrant juice. The applied low temperature (30 °C) in RO filtration preserved the valuable components (anthocyanins, phenols, acids, etc.) and the antioxidant capacity during the process. The initial solid content of the juice increased from 8 to $10^0$ to 22-$25^0$Brix in the RO concentrate. Concentrations of different physicochemical properties investigated by Bánvölgyi et al. (2009) are shown in Table 6. Microfiltration of umbu juice was able to eliminate particles in suspension and reduce pectin in permeate, producing a free of haze clarified juice. Protein was partially concentrated, and also a slightly increase of sugar content was observed in retentate (Ushikubo et al., 2007).

**Table 4**
Analyses of poliphenols and flavonoids in samples of pomegranate juice clarified by PEEKWC and PSU HF membranes (Cassano et al., 2015).

| Membrane type | Sample | Polyphenols (g/L) | Flavonoids (mg/L) | Total soluble solids (°Brix) | Suspended solids (%w/w) |
|---|---|---|---|---|---|
| PEEKWC | Feed | 1.576 ± 0.03 | 708 ± 14.1 | 16.0 ± 0.10 | 4.9 ± 0.09 |
| | Permeate | 1.062 ± 0.02 | 471 ± 9.4 | 15.4 ± 0.25 | 0.0 |
| | Retentate | 2.054 ± 0.04 | 738 ± 14.7 | 16.3 ± 0.09 | 6.3 ± 0.12 |
| PSU | Feed | 1.571 ± 0.03 | 741 ± 14.8 | 16.0 ± 0.28 | 4.9 ± 0.09 |
| | Permeate | 1.177 ± 0.02 | 562 ± 11.2 | 15.4 ± 0.09 | 0.0 |
| | Retentate | 1.702 ± 0.03 | 786 ± 15.7 | 16.0 ± 0.16 | 6.9 ± 0.13 |

**Table 5**
Physiochemical properties of untreated and clarified must at different transmembrane pressures ($T$ = 15 °C; $Q_f$ = 440 L/h) (Cassano et al., 2008).

| Sample | Color (%$A_{420}$) | Clarity (%$T_{625}$) | TSS (°Brix) | pH | Total phenolics (mg/L) | Acidity (% tartaric acid) |
|---|---|---|---|---|---|---|
| Feed | 0.301 | 70.95 | 19.3 | 3.0 | 1627 | 0.75 |
| Clarified must (20 kPa) | 0.180 | 81.84 | 18.1 | 2.81 | 1256 | 0.84 |
| Clarified must (40 kPa) | 0.184 | 83.36 | 17.9 | 2.82 | 1244 | 0.87 |
| Clarified must (60 kPa) | 0.184 | 83.94 | 17.7 | 2.83 | 1207 | 0.85 |
| Clarified must (100 kPa) | 0.160 | 4.13 | 17.5 | 2.85 | 1170 | 0.87 |
| Clarified must (130 kPa) | 0.159 | 83.75 | 17.5 | 2.86 | 1133 | 0.87 |

**Table 6**
Concentrations of antioxidant capacity, acids, total phenolics and anthocyanins in the permeate and retentate (Banvolgyi et al., 2009).

| | Antioxidant capacity (mmol AS/L) | Acid content (%) | Total phenol content (mg/L) | Anthocyanin content (mg/L) |
|---|---|---|---|---|
| Initial juice | 21.76 | 0.42 | 2.51 | 1.79 |
| UF retentate | 19.28 | 0.42 | 2.95 | 1.88 |
| UF permeate | 11.12 | 0.32 | 0.82 | 0.47 |
| RO retentate | 17.20 | 0.83 | 1.49 | 0.61 |
| RO permeate | 0.06 | 0.005 | 0.02 | 0.0033 |

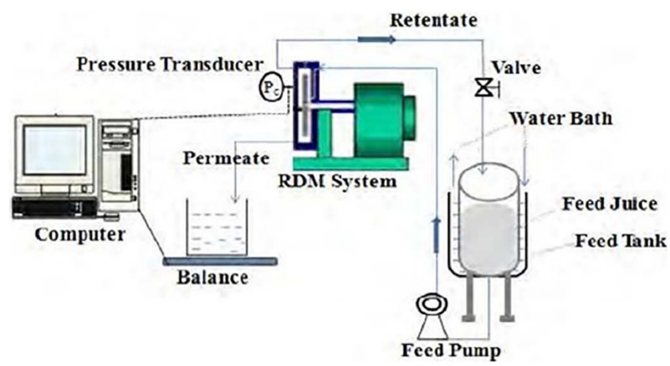


**Fig. 5.** Schematic diagram of the experimental set-up (Zhu et al., 2013).

### *5.4. Effect of membrane module in overall process efficiency and juice quality*

Membrane configuration, i.e., membrane geometry and the way it is mounted and oriented in relation to the flow of fluid, is crucial in determining the overall process performance. The most used configurations for the clarification of fruit juices at industrial level are tubular (inner diameter 5–10 mm), capillary (1–1.5 mm) and plate-and-frame membrane modules. Tubular and spiral wound membrane modules have an advantage over plate and frame membrane modules as they offer efficiency of uniform flow through the lumen, allowing industrial scale-up (Curcio et al., 2001). He et al. (2007) used a rectangular design plate and frame cross-flow membrane unit for apple juice clarification and found several advantages over tubular system such as lower capital and operating cost, higher flux rate due to the unique design of the plate etc. Layal et al. (2015) developed a lab scale dead end filtration module for fouling study in orange juice clarification and achieved good results compared to a tubular module. De Barros et al. (2003) studied cross-flow UF of depectinized pineapple juice using a tubular ceramic membrane (0.01 μm) and polysulfone hollow fiber membrane (100 ka). While comparing the fluxes for both modules, they found that the tubular module provided higher flux as compared to that of the hollow fiber membrane module. This can be described by the fact that the flow in turbulent regime in the tubular module increases the rate of solute diffusion from the membrane surface to the bulk which leads to less compact cake, causing a higher permeate flux compared with the flow obtained due to cake formation and the laminar flow in hollow fiber membrane. The promotion of a high degree of turbulence, high fluxes, low energetic costs per water volume unit, ease of operation and cleaning, as well as the possibility of scale up are determining factors for using a tubular membrane module in fruit juice clarification (Carvalho et al., 2008). De Oliviera et al. (2012) achieved significantly higher flux in tubular module compared to hollow fiber module for MF of passion fruit juice. Zhu et al. (2013) compared dead-end filtration with a rotating disk module for chicory juice clarification and observed that the former had showed the advantage of higher permeate flux and lower permeate turbidity, resulting from high membrane shear rate due high rotating speed, which also reduced membrane fouling and led to higher carbohydrate transmission. The experimental setup of Zhu et al. (2013) incorporating rotating disc module is shown in Fig. 5. Sarkar et al. (2009) used an electro-ultrafiltration system to analyze the effect of external d.c. electric field on the permeate flux. They observed a flux enhancement of 35.8% by applying an electric field of 400 V/m for fixed values of velocity and trans-membrane pressure and clarity of the juice also improved significantly. In apple juice concentration using nanofiltration, Warczok et al. (2004) found that irreversible fouling in flat sheet membranes was 68% higher than in tubular membrane. They also concluded that for long term applications, tubular membrane modules are more appropriate because of their longer lifetime and easier membrane recovery.

## 6. Fouling analysis/characterization in fruit juice clarification/concentration

A major limitation in applying MF and UF for fruit juice processing is the permeate flux decline due to the concentration polarization and membrane fouling (pore blocking and cake layer). While the cake layer formation due to concentration polarization is a reversible phenomenon, pore blocking is generally irreversible. A better understanding of the mechanisms of flux decline is critical for fouling control. Pore blocking methods based on Hermia models (Hermia, 1982) are used by several researchers to quantify irreversible fouling in fruit juice processing by membrane processes (Hojjatpanah et al. 2011; Nandi et al. 2012; Rai et al., 2007; Cassano, Donato et al., 2007; Cassano, Marchio et al., 2007; Mirsaeedghazi, Emam-Djomeh, Mousavi, Aroujalian, & Navidbakhsh, 2009; De Barros et al., 2003). In MF of passion fruit juice, De Oliveira et al. (2012) found that internal pore blocking predominated for ceramic tubular membrane, while cake filtration dominated for the hollow fiber membrane. Nandi et al. (2012) found cake filtration to be the dominant fouling mechanism during the filtration of centrifuged and enzymatic treated orange juice by using Hermia models. Rai et al. (2007) also found gel layer formation having the main role in flux decline during the crossflow ultrafiltration of depectinized mosambi juice. Similar results of cake formation were found by Mirsaeedghazi et al. (2009) by using Hermia models as shown in Fig. 6. This can be attributed to the large content of soluble solids found in fruit juices which contributes to the formation of a cake layer. Barros et al. (2002) reported that cake formation was the major fouling factor during the ultrafiltration of enzyme treated pineapple juice with polysulfone hollow fiber membranes. Standard pore blocking was found to be the major mechanism in MF of black mulberry juice clarification

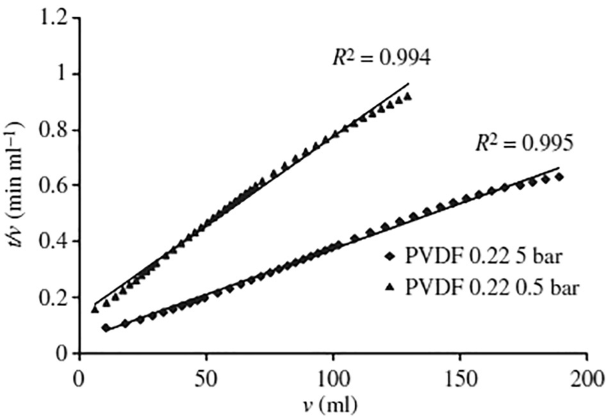


**Fig. 6.** The curve of t/v vs. v at different pressures in PVDF 0.22 μm (Mirsaeedghazi et al., 2009).

by mixed cellulose ester membrane of pore sizes 0.1 and 0.22 μm (Hojjatpanah et al., 2011). Scanning electron microscope (SEM) technique has been used by researchers to observe fouling in pores (Saha, Balakrishnan, & Ulbricht, 2007; Mirsaeedghazi, Emam-Djomeh, Mousavi, Ahmadkhaniha, et al., 2010; Mirsaeedghazi, Emam-Djomeh, Mousavi, Aroujalian, et al., 2010; Hojjatpanah et al. 2011; Warczok et al., 2004). Mathematical modeling results of pore blocking and cake formation of Hojjatpanah et al. (2011) and Mirsaeedghazi et al. (2009) were also validated by SEM analysis. Saha et al. (2007) was able to observe and analyze polysaccharide fouling in sugarcane juice UF by SEM images as shown in Fig. 7. Blocked pores and a layer of deposited particles were observed by Warczok et al. (2004) using SEM in nanofiltration membranes during apple juice concentration. Gulec, Bagci, and Bagci (2017) analyzed membrane fouling in ultrafiltration apple juice. They measured surface roughness of membrane using atomic force microscope and from their results concluded that membranes with more hydrophobic and rougher surface had higher fouling capacity than the ones with hydrophilic and smooth surface.

Researchers have investigated the decline of permeate flux by resistance- in-series model to quantify reversible and irreversible fouling (de Bruijn et al. 2002; Cassano et al., 2008; Cassano, Donato et al., 2007; Tasselli et al., 2007; Nourbakhsh, Alemi, Emam-Djomeh, & Mirsaeedghazi, 2014; Rai et al., 2006). Nourbakhsh et al. (2014) analyzed the influence of pressure on fouling resistances during MF of red plum and watermelon juices and noticed that all resistances, including cake, reversible, and irreversible resistances, increased remarkably when the transmembrane pressure was increased. Contribution of the reversible fouling resistance was far more significant compared to irreversible one in UF of kiwifruit juice (Cassano, Donato et al., 2007). de Bruijn et al. (2002) and Tasselli et al. (2007), both found fouling resistance to be the major one compared to cake and membrane resistances in apple and kiwifruit juice clarification by UF respectively. Research of Cassano et al. (2008) concluded that intrinsic membrane resistance controlled the permeate flux in pressure controlled region while cake resistance was the dominating one in mass transfer controlled region.

Table 7 presents types of membrane modules, their properties and operating parameters used in membrane processing of fruit juices

## 7. Application of pervaporation and electrodialysis in juice processing

### 7.1. Aroma recovery using pervaporation

Membrane Processes due to their high selectivity and possibility of operation at moderate temperatures, presents a promising alternative

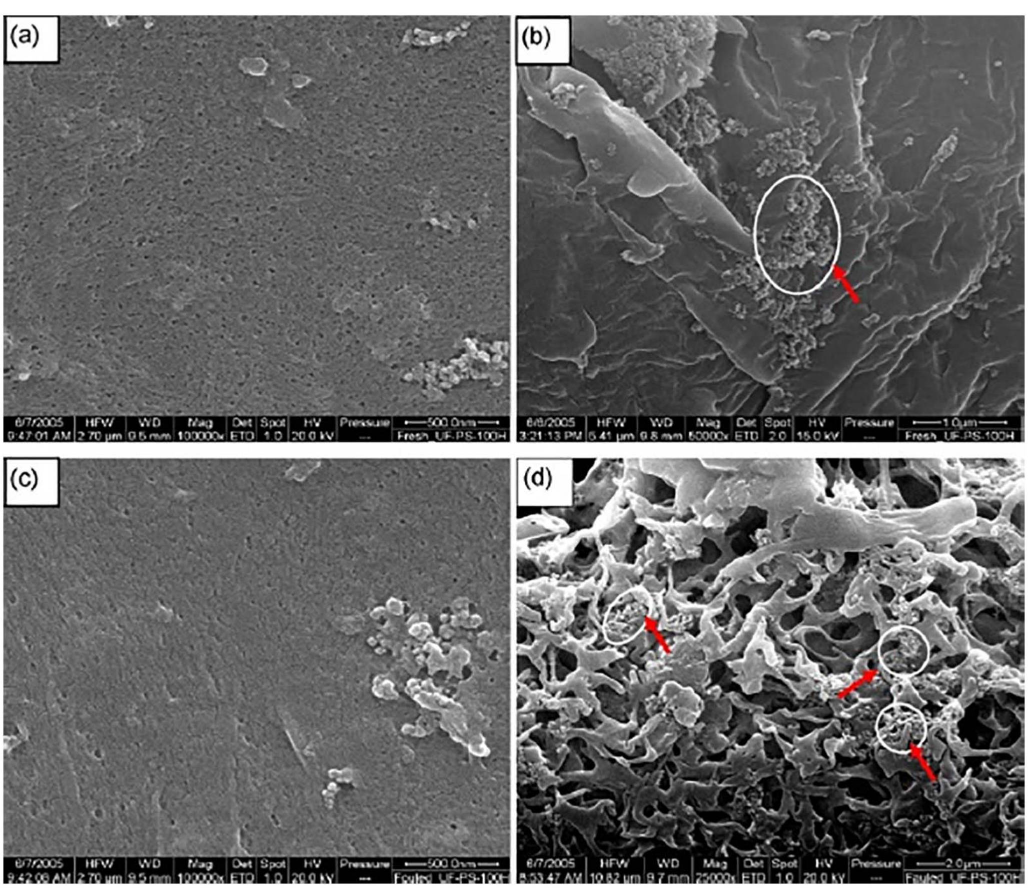


**Fig. 7.** SEM images of UF-PS-100H membrane: (a) pristine (surface), (b) pristine (cross-section), (c) polysaccharide fouled (surface) and (d) polysaccharide fouled (cross-section) (Saha et al., 2007).

**Table 7**
Literature on the types of membrane modules, their properties and operating parameters used in membrane processing of various fruit juices.

| Juice | Membrane | Optimal parameters TMP | CFV | Temp | Ref |
|---|---|---|---|---|---|
| Apple | Plate & frame PES, 50 kDa | 1–3 bar | 2.5 m/s | 50 °C | He et al. (2007) |
| Apple | Flat plate PES, 10 & 100 kDA | 1–3 bar | 0.1 m/s | 25 °C | Onsekizoglu et al. (2010) |
| Apple | Tubular Ceramic, 15 & 50 kDa | 1.5 bar | 7 m/s | 55 °C | Bruijn et al. (2003) |
| Apple | Tubular Ceramic, 50 & 300 kDa | 2 bar | 0.5 m/s | 20 °C | Vladisavljevic et al. (2003) |
| Apple | Tubular Polymeric, 0.3 μm | 2 bar | 30 °C | – | Aguiar et al. (2012) |
| Apple | Tubular PVDF, 18 kDa | 2.5 bar | 1 m/s | 50 °C | Yazdenshenas et al. (2005) |
| Orange | Plate & frame PVDF/PMMA, 0.2 μm | 1 bar | 1.25 m/s | 25 °C | Pagliero et al. (2011) |
| Orange | Tubular PVDF, 15 kDa | 1 bar | 1.5 m/s | 25 °C | Cassano, Marchio et al. (2007) |
| Orange | Flat plate PES, 30,50 & 100 kDa | 2 bar | – | 24 °C | Toker et al. (2013) |
| Pineapple | Tubular Polysulpone, 0.3 μm | 1.5 bar | – | 25 °C | Carvalho et al. (2008) |
| Pineapple | Tubular PES, 0.3 μm | 2 bar | 0.5 m/s | 25 °C | Carvalho et al. (2010) |
| Pineapple | Hollow fiber Polysulphone, 0.2 μm | 1 bar | 1.2 m/s | 20 °C | Laorko et al. (2010) |
| Lemon | Plate & frame PVDF, 0.2 μm | 0.8 bar | 1 m/s | 20 °C | Espamer et al. (2006) |
| Lemon | Tubular Ceramic, 15 kDa | 3 bar | 3 m/s | 20 °C | Maktouf et al. (2014) |
| Lemon | Hollow fiber PVDF, 0.45 μm | 1.2 bar | 1.5 m/s | 80 °C | Saura et al. (2012) |
| Melon | Tubular Ceramic, 0.2 μm | 2 bar | 7 m/s | 35 °C | Vaillant et al. (2005) |
| Melon | Plate & frame PVDF, 0.22 μm | 2 bar | 0.5 m/s | 50 °C | Nourbakhsh et al. (2014) |
| Melon | Tubular Ceramic, 0.1 μm | 3 bar | 6 m/s | 30 °C | Gomes et al. (2013) |
| Melon | Stirred cell Cellulose acetate, 0.2 μm | 2 bar | 1 m/s | 30 °C | Chhaya, Rai, Majumdar, Dasgupta, and De (2008) |
| Pomegranate | Plate and frame PVDF, 0.22 μm | 0.5 bar | – | 25 °C | Mirsaeedghazi, Emam-Djomeh, Mousavi, Aroujalian, et al., (2010) |
| Pomegranate | Tubular Ceramic, 0.2 μm | 3 bar | 4 m/s | 20 °C | Baklouti et al. (2011) |
| Pomegranate | Hollow fiber | 2.5 bar | 4 m/s | 25 °C | Cassano, Conidi, and Drioli (2011) |
| Grape | Hollow fiber Polysulphone, 100 kDa | 1 bar | 0.1 m/s | 35 °C | Cassano et al. (2008) |
| Kiwifruit | Tubular PVDF, 15 kDa | 0.5 bar | 1.4 m/s | 20 °C | Cassano, Donato et al. (2007) |
| Kiwifruit | Hollow fiber Polyetherketone, 50 kDa | 1 bar | 1 m/s | 25 °C | Tasselli et al. (2007) |
| Mosambi | Stirred cell Ceramic, 0.285 μm | 2.5 bar | 3 m/s | 25 °C | Nandi et al. (2009) |
| Mosambi | Stirred dead end cell Cellulose acetate, 0.2 μm | 1 bar | 1.2 m/s | 30 °C | Rai et al. (2006) |
| Passion fruit | Tubular Ceramic, 0.3 μm | 1 bar | 2 m/s | 25 °C | De Oliviera et al. (2012) |
| Sugarcane | Hollow fiber Polyamide, 0.4 μm | 1.2 bar | 1 m/s | 30 °C | Rezzadori et al. (2014) |
| Tomato | Flat frame Module PVDF, 0.45 μm | 1 bar | 1.5 m/s | 30 °C | Razi et al. (2011) |
| Umbu | Tubular Polypropylene, 0.2 μm | 2 bar | 0.5 m/s | 35 °C | Ushikubo et al. (2007) |

for aroma recovery from fruit juices (Karlsson & Tragardh, 1996). Since aroma compounds are highly heat sensitive, therefore high temperature thermal techniques are undesirable because of the loss of these compounds. Therefore pervaporation which enable separation of dilute species in liquid solutions offer a great scope for aroma recovery in beverage industry. Pervaopration has been already successfully applied to the recovery of the aroma compounds of several fruit juices such as apple (Olsson et al. 1999), kiwifruit (Cassano, Figoli, Tagarelli, Sindona, & Drioli, 2006), bergamot (Cassano, Conidi, & Drioli, 2013), strawberry (Isci, Sahin, & Sumnu, 2006), pineapple (Pereira et al., 2005), passion fruit (Pereira et al., 2005), banana (Sampranpiboon, Jiraratananon, Uttapap, Feng, & Huang, 2000), orange (Shepherd, Habert, & Borges, 2002) and grape (Rajagopalan et al. 1995).

Börjesson, Karlsson, and Trägårdh (1996) analyzed the efficiency of PDMS-1060, -1070 and PT1100 membranes for recovery of apple juice aroma. They were successful in getting enrichment factors upto 1000 with PDMS proving to be the most promising membrane material for such applications. In their research, they showed that the highest enrichment was provided by esters and the least was by alcohols. Bengtsson, Trägårdh, and Hallström (1989) and Alvarez et al. (2000) also concentrated various apple juice flavor compounds by using pervaporation and found same behavior. Isci et al. (2006) performed experiments for recovering strawberry aroma compounds with the help of pervaporation with binary, tertiary and multi-component solutions. Enrichment factors for methy and ethyl butanoate were comparatively same for binary and tertiary solutions while for multicomponent solutions, their results showed flux coupling adversely affecting the same. Pereira et al. (2005) studied pervaporation for aroma recovery of pineapple juice. They used single strength and clarified pineapple juices for investigation. The authors found very high enrichment of the most volatile components using composite ethylene–propylene-diene monomer (EPDM) hollow fiber. Their research concluded that when the organic solute concentration is reduced in the feed it is advantageous to choose a very selective polymer. Recovery of orange juice aroma components by pervaporation was studied by Aroujalian and Raisi (2007). They analyzed various parameters such as feed temperature, feed flow rate etc. on the overall efficiency of the pervaporation process. While increase in temperature helped in increasing flux and enrichment, feed flow rate didn't have much effect on both. They found highest enrichment factors for ethyl acetate while α-terpineol had the lowest value for the same. Raisi, Aroujalian, and Kaghazchi (2008) were successful in concentrating aroma compounds of pomegranate juice using pervaporation. They compared POMS and PDMS membranes for the same, while POMS fared better w.r.t. enrichment, PDMS showed more acceptable flux results. In their case also, increase in feed temperature resulted in higher flux and significant levels of enrichment. Shepherd et al. (2002) studied the use of PDMS hollow fibers in orange juice aroma recovery and found that temperature and feed flow rate had a positive effect on enrichment factors. Sampranpiboon et al. (2000) used POMS and PDMS membranes to recover aroma compounds from ethyl butanoate (ETB) and ethyl hexanoate (ETH) mixtures which are dominant mainly in pineapple and banana juice. Their results showed POMS membrane to be more permselective to the aroma compounds than the PDMS membrane. Hydrophobicity also played a role in the pervaporation performance, showing higher efficiency for the more hydrophobic ETH.

### 7.2. Deacidification of fruit juices using electrodialysis

The main application of electrodialysis in juice industry is deacidification. Fruit juices are valued by purchasers for their smell and flavors, yet the high acidity of some of them confines their utilization as a component in the formulation of various preparations such as beverages, ice creams, marmalades or cocktails (Vera et al., 2007b). The juice extracts from orange, grape, pineapple, and lemon are highly acidic. Acid concentrations of 1.0–1.2% in orange, grape, and pineapple juices interfere with utilization of these juices in single-strength or concentrated forms (Vera et al., 2009). Organic acids such as citric present in high concentration in lemon, orange, pineapple, and passion fruit juices or malic in apple and grape juices are responsible for fruit juices acidity. The sourness or sweetness in the juices is related to the ratio of soluble solids (sugars) to acids in the juice. In the juice industry, the ratio of soluble solids to acid in the juice is called the Brix/acid ratio. A high Brix/acid ratio is desirable in juices for taste and storage purpose. It can be effectively done by minimizing the acid content of the juice using electrodialysis.

Conventional electrodialysis was recommended for deacidifying several fruit juices such as orange, pineapple, grape (Adhikary, Harkare, Govindan, & Nanjundaswamy, 1983), mandarin orange (Kang & Rhee, 2002), and clarified passion fruit (Vera et al., 2003b) juices. Results of Kang and Rhee (2002) showed that total acidity was reduced by almost 30% with almost minimal changes in vitamin C and flavonoid content. Vera et al. (2003b) too found decrease in titratable acidity in case of passion fruit. They also found the acid strength to be decreasing which they associated with the conventional configuration enhancing the dissociation equilibrium of the weak citric acid. Vera et al. (2009) tested electrodialysis with bipolar membranes for removing citric acid from passion fruit juice. The authors found satisfactory results with deacidification up to a pH of 4.5 were achieved. The physicochemical properties were not affected in the process while a minimal color change was observed. After comparing both the conventional and bipolar electrodialysis (Vera et al., 2003a, 2003b, 2009) concluded that simultaneous production of organic acid in the bipolar system balances the higher equipment cost of the same than the conventional one. Therefore, electrodialysis with bipolar membranes could be an environmentally friendly alternative to the conventional techniques for the deacidification of fruit juices. Rozoy, Boudesocque, and Bazinet (2015) studied electrodialysis with bipolar membranes (EDBM) for deacidifying cranberry juice. Their experiment was successful in increasing the pH of the juice from 2.4 to 2.7 while deacidification rate was 22.84%. They also concluded that this rate can further be improved by increasing the number of membranes stacked in the module. Research of Serre, Rozoy, Pedneault, Lacour, and Bazinet (2016) found bipolar and anion-exchange membranes (ED2MB) most efficient for deacidification of cranberry juice with a rate of 40% in 3 h. They also recovered purified organic acids, or mixed organic acids in the recovery solution which can be used as preservative and or flavoring agents, in various food applications. The current efficiency for an electrodialysis system in the deacidification of fruit juice is from 52 to 90% depending on the quality of the juice. (Vera et al., 2007a, 2007b)

## 8. Membrane distillation in fruit juice concentration

Membrane distillation (MD) is a temperature driven process in which two liquid solutions, at different temperatures, are separated by a microporous hydrophobic membrane. Therefore, liquid-vapor interfaces are formed at the entrances of each pore. The hydrophobic nature of the membrane prevents penetration of the pores by aqueous solutions due to the surface-tension forces. In these conditions, a water-vapor transfer from the warm side to the cold one occurs. Vapor-pressure gradient between the two solutions separated by the membrane, generated by a temperature difference is the driving force of the process.

One of the prime benefits of MD is that it can be operated at atmospheric pressure and operating temperature is much lower than the boiling point of the solutions. Therefore it is very efficient in concentrating fruit juices which are sensitive to high temperature. Fixed costs and mechanical requirements on the membrane are greatly reduced due to lower pressure requirements. These features make MD ideal for the treatment of food and pharmaceutical solutions.

Although MD holds a good promise as an alternative to the present pressure-driven processes, it is still not fully commercialized in industrial setting due to the following issues:

- Lower permeate flux compared to RO
- Temperature polarization causing flux decay
- Module design
- Highly energy intensive

Table 8 shows some selected MD applications in fruit-juice processing: they refer to the concentration of clarified juices and to the recovery of aroma compounds by using DCMD and VMD configurations.

Drioli, Jiao, and Calabrò (1992) and Calabro et al. (1994) first studied the applicability of MD in fruit juice concentration by integrated membrane systems. They also considered the effect of viscosity and the necessity of juice pretreatment. Commercial plate PVDF membranes were used for the concentration of single-strength orange

**Table 8**
MD applications in fruit juice processing.

| Fruit juice | Membrane type | MD configuration | References |
|---|---|---|---|
| Apple | Enka Microdyn, hollow fiber, polypropylene | DCMD | Lagana, Barbieri, and Drioli (2000) |
| Apple (clarified by UF) | MFK3, flat sheet, PVDF | DCMD | Gunko, Verbych, Bryk, and Hilal (2006) |
| Blackcurrant (clarified by UF) | K150, flat sheet, PTFE | VMD | Bagger-Jorgensen et al. (2004) |
| Blackcurrant (clarified by UF & preconcentrated by RO) | Hollow fiber, polypropylene | DCMD | Kozak, Bekassy-Molnar, and Vatai (2009) |
| Orange juice (diluted from commercial concentrate) | Millipore, flat sheet, PVDF; Gelman, G0712,flat sheet; Enka, hollow fiber, polypropylene | DCMD | Calabro et al. (1994) |
| Pear (model solution) | Enka-Mycrodin, MD020TP 2 N, hollow fiber, polypropylene | VMD | Diban, Voinea, Urtiaga, and Ortiz (2009) |
| Apple (diluted from commercial concentrate) | Enka Microdyn, hollow fiber, polypropylene | DCMD | Curcio et al. (2005) |

**Table 9**
Main characteristics of the pineapple juices before and after concentration by osmotic evaporation (mean ± standard deviation on 2 trials) (Hongvaleerat et al., 2008).

| | Single strength juice | | | | Clarified juice | | | |
|---|---|---|---|---|---|---|---|---|
| | First stage | | Second stage | | First stage | | Second stage | |
| | Feed | Concentrate | Feed | Concentrate | Feed | Concentrate | Feed | Concentrate |
| Total soluble solids (g 100/g) | 12.6 ± 0.1 | 29.0 ± 1.4 | 31.3 ± 2.5 | 56.7 ± 1.8 | 10.6 ± 0.6 | 27.8 ± 0.4 | 30.2 ± 1.1 | 55.5 ± 3.5 |
| $a_w$ | 0.99 ± 0.01 | 0.97 ± 0.01 | 0.96 ± 0.00 | 0.90 ± 0.02 | 0.99 ± 0.01 | 0.98 ± 0.01 | 0.98 ± 0.01 | 0.92 ± 0.06 |
| pH | 3.77 ± 0.21 | 3.71 ± 0.31 | 3.70 ± 0.23 | 3.68 ± 0.29 | 3.96 ± 0.01 | 3.92 ± 0.02 | 3.88 ± 0.02 | 3.85 ± 0.02 |
| Titratable acidity (eq/L) | 0.83 ± 0.18 | 1.63 ± 0.32 | 2.38 ± 0.25 | 4.00 ± 0.14 | 0.70 ± 0.14 | 1.80 ± 0.42 | 2.23 ± 0.60 | 3.90 ± 0.99 |
| Total phenolic content (mg 100/g) | 18.6 | 46.3 | 61.3 | 106.2 | 4.9 | 42.9 | 47.2 | 112.3 |
| L | 34.33 ± 0.87 | 33.43 ± 0.23 | 33.08 ± 3.34 | 32.84 ± 5.30 | 29.96 ± 0.17 | 28.18 ± 0.61 | 28.77 ± 0.48 | 26.10 ± 1.16 |
| a | − 1.82 ± 0.54 | − 1.45 ± 0.23 | − 1.40 ± 1.15 | − 0.74 ± 0.11 | − 0.15 ± 0.19 | 0.44 ± 0.19 | 0.48 ± 0.21 | 1.57 ± 0.02 |
| b | 4.61 ± 1.03 | 5.65 ± 0.80 | 4.13 ± 1.68 | 3.35 ± 0.20 | 2.90 ± 0.30 | 4.11 ± 0.75 | 5.84 ± 0.27 | 2.76 ± 1.55 |

juice with a TSS content of $10.8^0$ Brix. The percentage reduction of flux was 50% when the juice was concentrated up to $31^0$ Brix at a transmembrane temperature gradient of $20^0$C. They found a very good retention of soluble solids, sugars and organic acids with rejection of sugars and organic acids equal to 100%. The observed reduction of vitamin C of about 42% was associated to high temperature and oxidation. They also found the color and flavor of the concentrated juice satisfactory. The pretreatment of the juice by UF helped in removing pulp and pectin which led to a clarified juice with a lower viscosity compared with the single-strength juice.

Researchers have shown that the combination of MD and pressure driven membrane separation processes offers important benefits over single use of MD in the concentration of various types of juices including grape juice (Rektor et al., 2006), pineapple juice (Hongvaleerat et al., 2008), kiwi fruit juice (Cassano & Drioli, 2007), camu-camu juice (Rodrigues et al., 2004), sugarcane juice (Nene et al. 2004) and cactus pear juice (Cassano, Conidi, et al., 2007). The effect of osmotic evaporation on components of single strength pineapple juice is shown in Table 9 (Hongvaleerat et al., 2008). Combination of MD with other membrane operations such as MF, UF, NF, RO and OD helps in achieving high quality fruit juice concentrates with higher economic feasibility. Kozak et al. (2009) performed pretreatment using enzymatic treatment, MF and RO of black currant juice and used DCMD in order to further concentrate the juice. Pretreatment increased the concentration from 15 to 22 °Brix. DCMD helped in concentrating further until 58 °Brix using a polypropylene membrane and a temperature difference of 19 °C (feed temperature 30 °C). For the parameters they analyzed, the concentrations were directly proportional to the increase of TSS in the juice, which indicated that retentions were quite high. Effect of temperature gradient on TSS content is shown in Fig. 8. Direct contact membrane distillation (DCMD) was applied for the concentration of apple juice using a polyvinylidene fluoride membrane by Gunko et al.

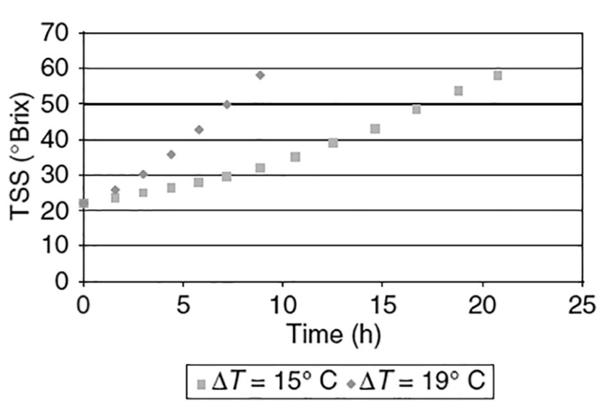


**Fig. 8.** TSS of the black-currant juice in function of time during the concentration with MD using different temperature differences (Kozak et al., 2009).

(2006). The pretreatment was done using an enzymatic step followed by clarification using ultrafiltration. A TSS content of 50 °Brix was obtained when the permeate flux reached about 9 L/m$^2$/h. Reduced productivity in terms of flux decay was observed beyond further concentration to 60–65 °Brix. Decreasing the cooling water temperature and maintaining a constant juice temperature in the hot cell had a significant effect on flux improvement. Similar flux enhancement was also noticed due to the pretreatment that lead to a lower viscosity and thus concentration and temperature polarization.

Lagana et al. (2000) produced highly concentrated apple juices up to 64 °Brix using polypropylene hollow-fiber DCMD modules with tube and shell configuration. They showed that flux rates were dependent essentially upon temperature polarization phenomena located mainly on the feed side, rather than concentration polarization which is insignificant. Temperature polarization becomes more prone to higher feed temperatures. Similar results were found by Curcio et al. (2000).

Bagger-Jorgensen, Meyer, Varming, and Jonsson (2004) illustrated the use of MD to recover and concentrate selectively volatile aroma compounds from black currant juice. The clarification step was performed using depectinization, clarified with gelatin-silica sol, centrifugation and finally ultrafiltration. In order to obtain a concentrated permeate, a vacuum membrane distillation (VMD) configuration was chosen and was performed at low temperatures (10–45 °C) by using a flat PTFE membrane. They observed that low temperature was suitable for obtaining highest concentration factors for the blackcurrant aroma compounds. They were a able to recover up to 83% of the components, whereby a maximum concentration factor 31 was obtained. Cane sugar concentration was done using DCMD by Nene, Kaur, Sumod, Joshi, and Raghavarao (2002). The initial feed concentration was 20 °Brix. They used a polypropylene membrane and a feed temperature of 70 °C. A pretreatment with microfiltration didn't resulted in flux improvement, which the author attributed to the higher polysaccharide content of the micro-filtered cane sugar fraction than the raw sugar which resulted in an increased concentration polarization. Quist-Jensen et al. (2016) tested DCMD for concentrating clarified blood orange juice. Their two step DCMD process was successful in achieving a final concentration of 65 °Brix. They also reported satisfactory levels of phenolics and antioxidant activity in the final product.

### 8.1. Effect of process parameters on membrane distillation

Permeate fluxes in MD are affected by different operating conditions such as: feed concentration, operating temperature, feed circulation velocity, temperature difference, permeate inlet temperature, flow velocity and vapor-pressure difference (Cassano & Drioli 2010). Permeate flux decreases with an increase in feed concentration (Souhaimi & Matsuura, 2011), which can be attributed to the reduction of the driving force due to declination of the feed vapor pressure. Increase of viscosity of the feed with increasing concentration also act as a factor (Onsekizoglu et al. 2015). The contribution of concentration polarization compared with temperature polarization effects is very minimal (Lagana et al., 2000). MD can be effectively applied for fruit juice concentration, where high retentate concentrations are desired as it can deal with highly concentrated feed solutions without suffering a major drop in permeability. This comes as an advantage over pressure driven membrane separation processes (Curcio & Drioli, 2005). Onsekizoglu et al. (2010) observed that the feed flow rate has minimal effect on transmembrane flux compared to temperature difference across the membrane during apple juice concentration using membrane distillation. The effect of flow rate on MD flux becomes more noticeable at higher temperatures especially associated with higher temperature drop across the membrane (Walton, Lu, Turner, Solis, & Hein, 2004). Increase in permeate flow and/or stirring rate helps in reducing the temperature polarization effect which ultimately leads to approaching of gas/liquid interface temperature to the bulk temperature at the permeate side, resulting in an increase in the driving force and MD flux (Hongvaleerat et al., 2008). In DCMD applications, increasing the permeate temperature is directly linked to a reduction in permeate flux which is attributed to the decrease of the transmembrane vapor pressure as long as the feed temperature is maintained constant (Lagana et al., 2000). Research of Quist-Jensen et al. (2016) correlated the evaporation flux decay in the pre-concentration step to the reduction of average temperature difference between the feed and permeate side while in the final concentration step, it was primarily affected by the increase in juice viscosity.

## 9. Forward osmosis (FO) concentration of fruit juices

Forward Osmosis comprises of a semi-permeable dense hydrophilic membrane, which separates two aqueous solutions (feed and draw solutions) having different osmotic pressures that acts as a driving force. The water transfer occurs from the feed side (low concentration) to the draw solution side (high concentration) across a semi-permeable membrane till the osmotic pressure difference between both sides is close to zero (Shaffer, Werber, Jaramillo, Lin, & Elimelech, 2015). The forward osmosis can be performed even at ambient pressure and temperature leading to the higher retention of thermo-labile components while the product can be concentrated up to higher concentration (up to 60 °brix) (Sant'Anna, Marczak, & Tessaro, 2012. In the fruit juice industry, compared to thermal concentration techniques, FO can help in achieving an acceptable level of physicochemical and sensory properties without deteriorating its quality (Petrotos et al. 2001).

Wrolstad et al. (1993) analyzed FO for concentrating raspberry juice using corn syrup as the draw solution. Their result showed that FO concentrated juice was strong in raspberry aroma and flavor and were comparable to some commercial samples. The final concentration of the juice was 45 °Brix. Herron, Beaudry, Jochums, and Medina (1994) used fructose (74 °Brix) as osmotic agent for concentrating orange juice using FO. They obtained a maximum osmotic flux of 4 kg/m$^2$/h and the quality of the final concentrated juice was superior when compared to the one concentrated by thermal evaporation. Petrotos, Quantick, and Petropakis (1998) successfully used FO for concentrating UF clarified tomato juice upto 52 °Brix using NaCl as draw solution. Babu, Rastogi, and Raghavarao (2006) used forward osmosis using mixed osmotic agent (sucrose 40% (w/w) and sodium chloride 12% (w/w)) for the concentration of pineapple juice up to 60 °Brix. The sucrose–sodium chloride combination was able to overcome the drawback of sucrose (low flux) and sodium chloride (salt migration) as single osmotic agents during direct osmosis process. They found satisfying results regarding the characteristics of the juice as shown in Table 10. Garcia-Castello et al. (2011) investigated the potential of FO for concentrating orange liquor solution using sodium chloride as draw solution. The TSS of the liquor increased from 8 to 10.5 °Brix. Their research showed that pectin was the main component that causes fouling, resulting in substantial decline in flux. Nayak, Valluri, and Rastogi (2011) concentrated pineapple and grape juice using FO. For the grape juice, anthocyanins and TSS were concentrated from 105 to 715 mg/L and from 8 to 54.6 °Brix, respectively while for pineapple juice, the TSS concentration increased from 4.4 to 54 °Brix. Shalini et al. (2016) concentrated raw sugarcane juice from 17.6 °Brix to 31.7 °Brix by using forward osmosis. They found better results w.r.t. color and flavor when compared to thermal evaporation.

**Table 10**
Comparison of physico-chemical characteristics of pineapple juices (Babu et al., 2006).

| Characteristic | Fresh juice | DO juice concentrate | Reconstituted juice |
|---|---|---|---|
| pH | 3.62 ± 0.10 | 3.85 ± 0.10 | 3.70 ± 0.10 |
| Titratable acidity (%, w/w citric acid) | 0.80 ± 0.10 | 2.50 ± 0.20 | 0.75 ± 0.10 |
| Ascorbic acid (mg/100 ml) | 12.50 ± 1.0 | 45.0 ± 2.0 | 12.10 ± 1.0 |
| °Brix | 12.40 ± 0.10 | 60.0 ± 0.20 | 12.4 ± 0.20 |
| Density (kg/m3) | 1060 ± 2.0 | 1260 ± 4.0 | 1070 ± 4.0 |
| Viscosity (mPa s) | 1.40 ± 0.20 | 35 ± 2.0 | 1.60 ± 0.20 |
| NaCl concentration (%) | – | 1.70 ± 0.02 | 0.58 ± 0.02 |
| $L^*$ | 40.18 ± 1.0 | 25.34 ± 1.0 | 39.14 ± 1.0 |
| $a^*$ | 3.64 ± 0.6 | 7.84 ± 0.6 | 3.87 ± 0.3 |
| $b^*$ | 19.83 ± 1.0 | 13.49 ± 1.0 | 19.44 ± 1.0 |
| Hue angle ($H^0$) | 79.60 ± 1.2 | 59.84 ± 0.06 | 78.74 ± 0.3 |
| Color purity ($C^0$) | 20.17 ± 1.1 | 15.60 ± 1.2 | 19.82 ± 1.1 |

## 10. Integrated membrane processes for juice concentration

Membrane-based approaches can provide an essential contribution to the concept of process intensification, which may lead to even larger performance benefits with regard to the fruit juice processing industry. The integration or the substitution of various traditional operations with innovative membrane based techniques permits the rationalization of direct and indirect energy consumption improving at the same time the organoleptic properties of the finished product. Integration of these membrane methods can help in reducing constraints, waste generation (Stankiewicz et al. 2000) and energy consumption which will improve economic viability of these processes (Alvarez et al., 2000; Sotoft, Christensen, Andrésen, & Norddahl, 2012). A general flow sheet of an integrated membrane process for the clarification and concentration of fruit juices is illustrated in Fig. 9. The process includes a preconcentration step based on the use of RO membranes followed by a final concentration by OD.

In recent years, researchers have analyzed integration of numerous membrane based methods for processing various fruit juices. Alvarez et al. (2000) used an enzymatic membrane reactor (EMR) for clarifying apple juice and RO for preconcentration and aroma recovery from the preconcentrated juice and a final concentration step up to 72 °Brix by using conventional evaporation. They were successful in retaining a high percentage of sugar and polyphenols in the preconcentrated juice and also achieved a high level of aroma enrichment. Cassano, Jiao, and Drioli (2004) investigated integrated membrane process for the production of kiwi fruit juice concentrate. They performed UF for clarification and OD for concentration. They achieved a final concentration of 60 °Brix and a high percentage of vitamin C was retained in the concentrate. Cisse, Vaillant, Perez, Dornier, and Reynes (2005) used MF for clarifying orange juice and used OD for concentrating the clarified juice. They were able to concentrate the juice upto 62 °Brix with minimal declination of evaporation flux. Koroknai, Csanádi, Gubicza, and Bélafi-Bakó (2008) used UF for clarifying three types of red juices (chokeberry, redcurrant and cherry) and coupled MD and OD for further concentration. Because of this coupling of MD and OD, they achieved an enhanced water flux due to an increase of the driving force. They were able to retain a high percentage of antioxidant activity in the product juice. This coupled operation of MD and OD produces more

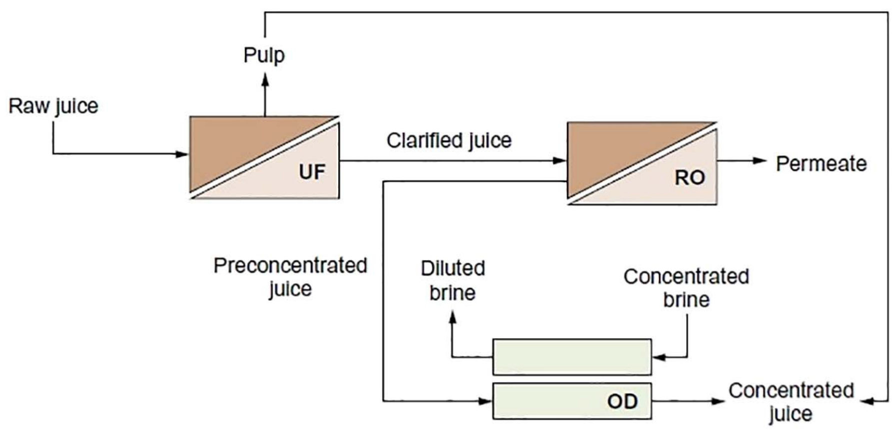


**Fig. 9.** Flow sheet of integrated membrane process for the clarification and concentration of fruit juices.

efficiency than single use of any of them (Bélafi-Bakó & Koroknai, 2006). Cassano et al. (2013) used UF and OD for clarification and concentration of bergamot juice and found that the concentrated juice was able to retain antioxidant properties of the fresh juice. Integrated membrane processes have also been analyzed for the clarification and concentration of tropical juices such as camu-camu (Souza et al., 2013) and acerola (Pagani et al., 2011) and were successful in concentrating the juices upto a TSS content of 53–55 °Brix after final concentration with OD with no losses of vitamin C content and antioxidant activity. Sotoft et al. (2012) used RO/NF, DCMD and VMD for preconcentration, final concentration and aroma recovery respectively of blackcurrant juice. By combining NF with RO, they were able to overcome the high osmotic pressure limitations presented by RO. The DCMD step was able to concentrate the juice upto 70 °Brix. Chaparro et al. (2016) used combination of enzymatic maceration, microfiltration, diafiltration and centrifugation to extract and purify lycopene from watermelon juice. Their integrated process obtained a natural extract of lycopene that was 41 times more concentrated and 34 times purer than the initial juice, yielding an extract with up to 2% all-trans-lycopene on a dry basis. Oliviera et al. (2016) coupled microfiltration, diafiltration and reverse osmosis and were successful in concentrating lycopene and partitioning the sugars present in watermelon juice in order to obtain a novel extract. The lycopene content of the product juice was 17.7 times higher than the fresh juice. Antioxidant capacity also increased and the sugar content decreased due to the difiltration step. Carotenoids profile and color parameters of the reversed osmosis extract is listed in Table 11.

## 11. Economic feasibility of integrated membrane processes

Researchers have analyzed economic feasibility of integrating various membrane methods for fruit juice processing and found positive results. Molinari, Gagliardi, and Drioli (1995) used substitution coefficient (CS) for calculating energy savings in membrane operations. Substitution coefficient is defined as the ratio between the primary energy (thermal) saved in the new process with respect to the conventional process and the amount of electrical energy consumed, relative to the conventional process and is mathematically defined as

$$CS = \frac{C1 - C2}{E2 - E1}$$

where $C$ is the consumption of thermal primary energy (MJ or Mcal), $E$ is the consumption of electrical energy (kWh), and 1 and 2 are the relative indexes of the conventional and innovating process, respectively. The author found high values for energy savings in membrane processing of tomato (CS = 137.9 MJ/kWh) and orange (CS = 52.5–766.5 MJ/kWh) juice when compared with thermal evaporation. An economic evaluation made by Alvarez et al. (2000) of the integrated membrane system for apple juice concentration indicated a reduction of the total capital investment of 14% and an increase in process yield of 5% when compared with the conventional process. Total manufacturing costs decreased by 8% because less energy was required to concentrate the juice. Membrane replacement accounted only for 2% of operating costs and membrane life was estimated to be 2, 3 and 2 years for UF, RO and PV membranes, respectively. The cost comparison of conventional and integrated membrane process is shown in Table 12. For black currant juice concentration, Sotoft et al. (2012) used integrated membrane systems comprising of NF, RO, DCMD and VMD for aroma recovery. They found that the estimated operation cost is lower than the price of a traditional process by about 43% and concluded that the economical potential of the process is very promising in order to replace conventional evaporators. Hogan, Canning, Peterson, Johnson, and Michaels (1998) reported a total process cost of OD concentration in the order of $1.00/l of concentrate. From 1 L of fresh juice, it is possible to achieve about 200 ml of 70 °Brix concentrate. The value of this concentrate is between $2.50 and $7.50/L. From these data, the economical benefits of the integrated membrane process seem evident.

**Table 11**
Carotenoids profile and color parameters of the reversed osmosis extract[1] (Oliveira et al. 2016).

| Analysis | Concentrated extract |
|---|---|
| Total carotenoids (μg/g) | 808.07 ± 39.89 |
| β-Carotene (μg/g) | 50.69 ± 0.01 |
| Lycopene (μg/g) | 723.45 ± 38.33 |
| L* | 37.09 ± 0.17 |
| a* | 21.14 ± 0.26 |
| b* | 7.72 ± 0.15 |
| C* | 22.50 ± 0.30 |
| h | 20.06 ± 0.14 |

[1] = Mean of 3 determinations.
[1] = Carotenoids determined by HPLC analysis.

## 12. Major membrane suppliers in the juice industry

There are numerous membrane and membrane module manufactures and suppliers around the world. The main suppliers for the juice industry are Alfa Laval, GEA Filtration, GE Osmonics, Ionics Inc., Koch Membrane Systems, Pall Corp., Sartorius AG etc.

## 13. Critical observations

Pretreatment of pectin containing juices using enzymatic treatment helps in minimizing gel/cake layer formation and subsequently fouling in membrane based clarification processes. Microfiltration can be used as an effective pretreatment technique for further concentration using RO and membrane distillation methods. Microfiltration and Ultrafiltration are very successful in removing suspended solids, hence

**Table 12**
Capital investment and manufacturing costs for the conventional and integrated membrane process (Alvarez et al., 2000).

| Item | Cost (EURO) | |
|---|---|---|
| | Conventional process | Membrane process |
| Investment | | |
| Apple storage | $1.7 \times 10^5$ | $1.7 \times 10^5$ |
| Washing and inspecting apples | $0.1 \times 10^5$ | $0.1 \times 10^5$ |
| Milling apples | $0.4 \times 10^5$ | $0.1 \times 10^5$ |
| Clarification | $6.4 \times 10^5$ | $3.9 \times 10^5$ |
| Preconcentration (RO) | | $1.3 \times 10^5$ |
| Aroma compounds recovery and apple juice concentration | $4.2 \times 10^5$ | $5.1 \times 10^5$ |
| Total | $13.73 \times 10^5$ | $12.48 \times 10^5$ |
| Total capital investment | $47.72 \times 10^5$ | $43.38 \times 10^5$ |
| Operating costs | | |
| Raw material (apples) | 810.0 EURO/ton conc. | 810.0 EURO/ton conc. |
| Membranes | | 19.7 EURO/ton conc. |
| Others | 99.9 EURO/ton conc. | 50.2 EURO/ton conc. |
| Total variable cost | 909.9 EURO/ton conc. | 839.9 EURO/ton conc. |
| Labor, maintenance and other fixed costs | 151.9 EURO/ton conc. | 145.0 EURO/ton conc. |
| Total manufacturing costs | 1068.8 EURO/ton conc. | 984.9 EURO/ton conc. |

turbidity of fruit juices. They improve clarity of juices significantly. Soluble solids content in the clarified juices is not much affected by both MF and UF and the slight decrease can be attributed to the removal of suspended solids. Various authors have reported a higher value of soluble solids in the retentate side during MF and UF of fruit juices and linked it to the presence of high suspended solids content in the pulpy products, interfering with the measurement of refractive index. Acid content measured as titratable acidity and pH largely remains unaffected in juices clarified by MF and UF. Ascorbic acid loss has been observed in RO and UF of fruit juices which can be associated with the possible occurrence of oxidative reactions during concentration, as these processes run for a long time. Loss of phenolic compound is also noticed in some cases, but is minimal compared to antioxidants. Flux decline with time due to fouling remain one of the main challenges for fruit juice processing using membrane technology, though various flux enhancement techniques such as use of turbulence promoters, gas Sparging, back flushing, application of ultrasonic and electric field, shear enhanced modules etc. are successful in mitigating fouling affects. Low evaporation fluxes in MD seem to be the main drawbacks when compared with RO and thermal evaporation. Theoretical 100% rejection to nonvolatile solutes is one of the major advantages of membrane distillation. For fruit juice concentration, recent research shows that coupled operation of MD and OD can favourably deal with high temperature related problems (i.e. aroma and color loss) prevalent in MD. Also, utilization of low-grade waste and/or alternative energy sources for MD can be another promising area of research due to its ability to effectively operate at low temperatures. For Forward osmosis, recovery and regeneration of the draw solution can be construed as the one of the most significant limitations in transforming forward osmosis into a full-scale process in the juice industry.

## 14. Conclusion

The demand for healthy food products from diet and fitness conscious consumers is one of the leading drivers of the global juice market. Thus, the global market for juice is likely to witness strong growth over the forthcoming years. The sustained expansion of this market is associated with growing opportunities for fruit juice processing industries and food scientists to develop innovative methods in order to retain as much as possible the originality of fresh fruit w.r.t. color, aroma, nutritional value and structural characteristics. The potential of membrane technologies in the food and beverage industries is widely recognized today. One of the challenges of membrane technology for juice processing is scale up of laboratory modules to pilot plant studies, although some recent research has been done, but long term suitability has not been evaluated. Minimization of waste generated is another area which promises some research, although membrane methods generate very low waste compared to traditional methods. Safety aspects regarding stability and transport are also some important factors which needs attention.

Some of the main issues hindering the expansion of membrane operations for juice processing, such as MF and UF in the clarification of fruit juices, are basically related to membrane fouling. Fouling results in decrease of permeate flux and also reduces membrane life span. Innovation in cleaning methods can help in wide applicability of these methods in the juice industry. Surface modification of membranes is another area which can enable low fouling membranes to come into the picture. Ceramic membranes provide some advantages over polymeric membranes, but they are limited to small-scale applications because of their high costs, although research is going on for developing low cost ceramic materials and their applicability for juice processing. Development of new antifouling materials for membrane, module design, novel cleaning methods are some important areas of investigation for the sustainable growth of membrane operations in the clarification/concentration of fruit juices and beverages.

## Abbreviations

| | |
|---|---|
| UF | ultrafiltration |
| MF | microfiltration |
| MWCO | molecular weight cut off |
| SS | suspended solids |
| RO | reverse osmosis |
| OD | osmotic distillation |
| MD | membrane distillation |
| NF | nanofiltration |
| DE | diatomaceous earth |
| ED | electrodialysis |
| TMP | transmembrane pressure |
| VCF | volume concentration factor |
| TSS | total soluble solids |
| OE | osmotic evaporation |
| PVDF | polyvinylidene fluoride |
| TAC | total anthocyanin content |
| AA | ascorbic acid |
| TAA | total antioxidant activity |
| PES | polyethersulphone |
| HPLC | high performance liquid chromatography |
| DPPH | 2, 2-diphenyl-1-picrylhydrazyl |
| SEM | scanning electron microscope |
| PEEK | poly (ether ether ketone) |
| PTFE | polytetrafluoroethylene |
| PP | polypropylene |
| DCMD | direct contact membrane distillation |
| VMD | vacuum membrane distillation |
| PV | pervaporation |
| FO | forward osmosis |

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