



## Critical Reviews in Food Science and Nutrition

Publication details, including instructions for authors and subscription information:

<http://www.tandfonline.com/loi/bfsn20>

### Application of Membrane Separation in Fruit and Vegetable Juice Processing: A Review

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Accepted author version posted online: 20 Sep 2013.



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To cite this article: Susmit Ilame & Satyavir Singh (2013): Application of Membrane Separation in Fruit and Vegetable Juice Processing: A Review, Critical Reviews in Food Science and Nutrition, DOI: [10.1080/10408398.2012.679979](https://doi.org/10.1080/10408398.2012.679979)

To link to this article: <http://dx.doi.org/10.1080/10408398.2012.679979>

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## Application of Membrane Separation in Fruit and Vegetable Juice Processing: A Review

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### ABSTRACT

Fruit and vegetable juices are used due to convenience. The juices are rich in various minerals, vitamins, and other nutrients. To process the juices and their clarification and / or concentration is required. The membranes are being used for these purposes. These processes are preferred over others because of high efficiency and low temperature. Membranes and their characteristics have been discussed in brief for knowing suitability of membranes for fruit and vegetable juices. Membrane separation is low temperature process in which the organoleptic quality of the juice is almost retained. In this review, different membrane separation methods including Microfiltration, Ultrafiltration and Reverse osmosis for fruit juices reported in the literature are discussed. The major fruit and vegetable juices using membrane processes are including the Reverse osmosis studies for concentration of Orange juice, Carrot juice and Grape juice are discussed. The Microfiltration and Ultrafiltration are used for clarification of juices of mosambi juice, apple juice, pineapple juice and kiwifruit juice. The various optimized parameters in membranes studies are pH, TAA, TSS, AIS. In this review, in addition to above the OD is also discussed, where the membranes are used.

*Keywords: - Fruit juice, membrane separation, microfiltration, ultrafiltration, reverse osmosis, diafiltration and osmotic distillation.*

## 1 INTRODUCTION

Fruit and vegetable juices are valuable source of vitamins, minerals and other nutrients. There is a steady rise in consumption of processed juice. Currently the consumption of different fruit and vegetable juice is about 46.8 Billion liters by 2012 (marketpublishers.com). This is having a market value around 4,650,00US\$. However, after extraction juice processing involves filtration, clarification, concentration and spray drying etc. By filtration of bigger particles/ pulps are separated. The concentration is mostly carried out in evaporators or freeze concentration. However, for clarification and concentration of juice, the use of membranes reported the literature has been reviewed in this paper.

Commercially membranes are being used with the following juices such as Orange juice, Mosambi juice by Infra Food Brands, Ltd. Aurangabad, India. Apple juice, Pineapple juice, grape juice by Del Monte Foods, Italy and Welch Food Inc, Indonesia.

Reverse osmosis [RO] and ultrafiltration [UF] are the most versatile separation processes in the food industry; RO is essentially a concentration in which water is separated from low molecular weight solutes. UF is a clarification or fractionation process in which smaller solutes are transported across the membrane along with water and the membrane retains only large molecules (e.g., proteins and colloids ), depending on the pore size of the membrane. Microfiltration (MF) is yet another membrane process, which is mainly used for clarification purpose due to large membrane pore size.

Fruit juice concentration can be described as a separation process and forms of the basic unit operations of fruit technology. Basically the types of concentration processes are Freeze concentration, in which two distinctive steps, viz. ice crystallization and ice separation from

concentrate phase are involved. In the first stage, fruit juice is super cooled below its freezing point to allow water to separate as ice crystals. In the second stage, the ice crystals are separated from the concentrated fruit juices. Several factors affect the ice – separation efficiency, the foremost being the viscosity of the slurry and the ice crystals diam (Vanpelt, 1981) and Evaporative concentration, is probably the oldest methods of concentration and even today, it is considered to be the best developed, economically the most favourable and widely used methods for the concentration of liquid foods. The choice of a proper evaporator for concentrating a given material is determined by many factors, which must be carefully weighed to ensure that the process requirements and capital cost are met. These factors include properties of feed material, quality requirements of the product, operating conditions and operating economy (Mehra, 1986).

To reduce the storage and shipping cost, and to achieve longer storage, fruit juice are usually concentrated by multi-stage vacuum evaporation but, this process results in a loss of fresh juice flavors, color degradation and a “cooked” taste due to thermal effects. Since consumers generally prefer the flavor, aroma, appearance and mouth feel of freshly squeezed juices. The juice industry has developed complex essence recovery, careful process control and blending techniques to produce a good quality concentrate that is acceptable to consumers. Many efforts have been devoted to develop improved methods such as Freeze concentration, Evaporative concentration and the most promising alternative is membrane concentration (Chen and Shaw, 1993).

Membrane separation method is performed because of high efficiency and low temperature. The concentration of fruit juices reduces the volume with consequent reduction of transport, storage and packaging costs (Niemi and Bulsari, 1997). In addition, the concentrates

are more stable, presenting higher resistance to microbial and chemical deterioration than the original juice as a result of water activity reduction. Since the water content of fruit juice is very high about (75% to 90%). Concentration of fruit juice not only provides the microbiological stability, but also permits economy in packaging and distribution of the finished product due to reduction in bulk by weight and volume (Labuza, 1970).

In all these methods appreciable changes in organoleptic quality of product occur but in membrane separation due to low temperature, the organoleptic quality of juice products is almost retained, therefore membrane separation process is preferred over others. Filtration through a membrane where a dispersion, called a feed dispersion, is separated into a retentate not passing through the membrane and a permeate passing through said membrane. The retentate is a fluid dispersion in which the amount of dispersing phase has been increased relative to the amount there of in the feed dispersion. (Saugmann et al., 1997).

## **2 MEMBRANES:-**

Membranes can be defined as semi permeable barriers that separate two phases and restrict the transport of various substances in a specific way (Strathmann, 1990). The primary function of a membrane is to act as a selective barrier, allowing the passage of certain components and the retention of others from a determined mixture, implying the concentration of one or more components both in the permeate and in the retentate. The barriers considered here do not prevent the passage of all species but are permeable to some and impermeable to others, such membranes are termed semi permeable and usually are in the form of thin sheets of polymeric materials. Since amount of a species transported across a membrane is inversely proportional to the thickness. It is advantageous to have the thinnest membrane possible. In

practice, considerations such as mechanical strength usually determine the lower limit of membrane thickness. In many cases synthetic polymers are used and many have been developed specially to provide the required semi permeable characteristics (Sammon, 1972). Its selectivity is related to the dimensions of the molecule or particle of interest for separation and the pore size, as also the solute diffusivity in the matrix and the associated electric charges (Cheryan, 1998). The separation performance of a membrane is influenced by its chemical composition, temperature, pressure, feed flow and interactions between components in the feed flow and the membrane surface (Lin et al., 1997). In membrane separation Retentate is the fraction of a feed dispersion which is retained by a filtration membrane and Permeate is the fraction of feed dispersion which passes through a filtration membrane.

## **2.1 MEMBRANE CHARACTERISTICS AND TYPES:-**

Membranes can be classified as symmetrical or asymmetrical according to structure. This asymmetry is considered with respect to the internal structure of the membranes. In symmetrical membranes uniform pore sizes present in their cross-section whereas the pores of asymmetric membranes are usually larger (Cheryan, 1991). Thickness, pore diameter, solvent permeability and porosity are the main characteristics of membrane. Other important parameters are: permeate flow rate, heat, chemical and mechanical resistance (Ostergaard, 1998).

Membranes can be divided into two large categories: dense and porous according to the morphological point of view. Membranes are considered to be dense when transport of the components involves a stage of dissolution and diffusion across the material constituting the membrane. On the other hand a membrane is denominated as porous when permeate transport

occurs preferentially in the continuous fluid phase which fills the membrane pores (Harbert et al., 2006).

Commercial synthetic membranes are produced from two distinct classes of material: polymers consisting of organic material such as: cellulose acetate (CA), polyamide (PA), polysulfone (PS) and polyvinylidene difluoride (PVDF) amongst others; and inorganic materials such as: metals and ceramic materials (Cuperus and Nijhuis, 1993). According to their technological evolution, membranes can be divided into four distinct classes:

First generation: membranes derived from cellulose acetate, originally developed for seawater desalination. They are sensitive to pH (3–8) and to temperature (maximum 50 °C), as well as being susceptible to microorganisms and disinfectants (Cheryan, 1998).

Second generation: membranes elaborated from synthetic polymers, mainly polysulfone or polyolefine derivatives. They were introduced as from 1975, showing variations in their chemical compositions and functional properties, such as those from polyamides and polysulfones. Polusulfone are easily spun into hollow fibers. They are resistant to hydrolysis (cleavage of the internal polymer bonds), to strong acids and bases and to high temperatures, but present low resistance to mechanical compacting. They are currently the most used membranes (Porter, 1990).

Third generation: membranes constituted of a ceramic material based on zirconium or alumina oxide, deposited on a surface made of graphite or other materials. They show great mechanical resistance and support high pressures. They also tolerate the entire pH range (0–14) and temperatures of over 400 °C and are chemically inert, but they are very costly (Cuperus and Nijhuis, 1993).

Fourth generation: these include recent developments in membrane technology resulting in a hybrid process consisting of a combination of conventional electrodialysis and membranes with different pore sizes, used in processes such as: microfiltration, ultrafiltration and nanofiltration (Aider et al., 2008). This continuous electrophoresis with porous membranes (CEPM) can be defined as an electrochemical process for the separation of charged organic molecules. The separation driving force of this process is based on the direct application of an electric field. Under the effect of an electric current, the ions are transported from one solution to another through one or more semipermeable porous membranes (Bazinet et al., 1998).

The main physical operational parameters that affect the permeate flow rate are: pressure, temperature, viscosity and density of the feed fluid, and the tangential velocity (Scott, 2003). The permeate flow rate is directly proportional to the pressure applied and inversely proportional to the viscosity. The viscosity can be controlled by two factors: solids concentration in the feed and temperature (Hwang and Kammermeyer, 1998). An increase in feed concentration alters the viscosity, density and diffusivity of the feed solution, causing a decrease in permeate flow rate (Satyanarayana et al., 2007). An increase in temperature results in a decrease in fluid viscosity and increase in molecular mobility, that is, in diffusivity. For its part, an increase in tangential velocity increases the permeate flow rate by provoking greater turbulence, causing a dispersion in the solute molecules concentrated on the membrane surface, reducing the thickness of the gel layer. This is one of the simplest and most effective methods to control the effect of concentration polarization (Cheryan, 1998; Strathmann, 1990). In membrane processes, an increase in pressure results in a greater convective rate for the transport of solute to the membrane surface, increasing its concentration at the interface, causing an increase in diffusivity



of the solute in the opposite direction to that of the process pressure, thus decreasing the permeate flow rate (Field et al., 1995)

There is a linear relationship between flow rate and the inverse of the solvent viscosity for nanofiltration and ultrafiltration membranes, indicating that the main mass transport mechanism in these systems is convection. In the majority of ultrafiltration membranes, this linear relationship between flow rate and the inverse of the solvent viscosity remains independent of the concentrations of organic solvents miscible with the aqueous solutions (Tsui and Cheryan, 2004). The configuration (flat sheet, tubular modules, hollow fibers, plates units, spiral wound) also affects membrane performance. Good membrane performance with respect to permeate flow rate and retention of the desired solute should be balanced with respect to its characteristics such as propensity for fouling, cost, ease of cleaning and substitution (Cheryan, 1998).

Membrane processes as microfiltration (MF), ultrafiltration (UF) and reverse osmosis (RO) have been widely applied to the dairy, food and beverage industry after the discovery of asymmetric membranes (Loeb and Souriragin, 1960).

#### **ADVANTAGES AND DISADVANTAGES OF MEMBRANE SEPARATION:-**

Membrane processes have a number of pluses and minuses compared to alternative means of performing separations (Yamamoto et al., 1989).

The advantages include:

- 1) Membrane processes can separate at the molecular scale up to a scale at which particles can actually be seen, this implies that a very large number of separation needs might actually be met by membrane processes (Cassano et al., 2007).

- 2) Do not require a phase change to make a separation (with the exception of pervaporation). As a result, energy requirements will be low unless a great deal of energy needs to be expended to increase the pressure of a feed stream in order to drive the permeating components across the membrane (Kilduff and Robeson, 1992).
- 3) Membrane processes present basically a very simple flow sheet. There are no moving parts (except for pumps or compressors), no complex control schemes, and little ancillary equipment compared to many other processes. As such, they can offer a simple, easy-to-operate, low maintenance process option (Leite, 2007).
- 4) A very large number of polymers and inorganic media can be used as membranes; there can be a great deal of control over separation selectivities (Dutta, 2007).
- 5) Membrane processes are potentially better for the environment since the membrane approach require the use of relatively simple and non-harmful materials (Harbert et al., 2006).

The disadvantages include:

- 1) Membrane processes seldom produce two pure products, that is, one of the two streams is almost always contaminated with a minor amount of a second component. In some cases, a product can only be concentrated as a retentate because of osmotic pressure problems. In other cases the permeate stream can contain significant amount of materials which one is trying to concentrate in the retentate because the membrane selectivity is not infinite (Carvalho et al., 2008).

- 2) Membrane processes cannot be easily staged compared to processes such as distillation, and most often membrane processes have only one or sometimes two or three stages. This means that the membrane being used for a given separation must have much higher selectivities than would be necessary for relative volatilities in distillation. Thus the trade-off is often high selectivity/few stages for membrane processes versus low selectivity/many stages for other processes (Johnson, 1996).
- 3) Membrane processes can be saddled with major problems of fouling of the membranes while processing some type of feed streams. This fouling, especially if it is difficult to remove, can greatly restrict the permeation rate through the membranes and make them essentially unsuitable for such applications (Liang, 2008).
- 4) Membrane modules often cannot operate at much above room temperature. This is again related to the fact that most membranes are polymer-based, and that a large fraction of these polymers do not maintain their physical integrity at much above 100 °C. This temperature limitation means that membrane processes in a number of cases cannot be made compatible with chemical processes conditions very easily (Labuzza, 1970).

### **3 MEMBRANE MATERIALS:-**

#### **3.1 Reverse osmosis:-**

Reverse osmosis (RO) or Hyperfiltration (HF) is a filtration method that removes many types of large molecules and ions from solutions by applying pressure to the solution, when it is on one side of the selective membrane and the result is that the solute is retained on the pressurized side of the membrane and the pure solvent is allowed to pass to the other side. RO

membrane mostly used in the clarification of orange juice, pear juice, passion fruit, carrot juice etc. (Leite, 2007).

The process of membrane technology becomes practical only with the development of suitable membranes. Most commercial RO membranes are usually made from cellulose acetate cast on asymmetric film. These membranes give excellent performance with respect to high permeate flow and high rejection of small molecules or ions; though these membranes have limited temperature and pH tolerance. These are susceptible to microbial and enzymatic attack. However, these disadvantages have been overcome by developing new non-cellulosic membranes. These membranes are most resistant to heat and chemical attack and hence most widely applicable in industrial processing (Sourirajan, 1978). In reverse osmosis process, it consists of High pressure pump followed by an Energy Recovery Device and the reverse osmosis membrane. At the start, the clarified juice is pressurized by the high pressure pump typically between 55 and 85 bars, depending on the temperature and the salinity of the juice. The pressure drop over the RO membranes is about 1.5 to 2 bar, depending on the number of element per pressure vessel, so the concentrate is released at high pressure. Because of energy recovery device, it is possible to reuse the energy from the concentrate flow. The concentrate is directed to the ERD, where it directly transfers its energy to part of the incoming feed. Specific energy required per kg of juice is  $3.5 \text{ kWh/m}^3$  (Westmoreland, 1968).

The potential of developing RO process as a concentration technique to remove water from fruit juice has been of interest to the fruit juice industry for about 30 years. The advantages of RO over traditional evaporation are in low thermal damage to product, reduction in energy consumption and lower capital investments (Merson and Paredes, 1980) as the process is carried

out at low temperature and it does not involve phase change for water removal. The retention of juice constituents, especially flavors, and the permeate flux, regarding RO performance, are two major factors, which are related to the type of membranes and the operating conditions used during the process. A considerable research has been carried out for the concentration of a variety of fruit juices, including apple, pears, grapefruit, kiwi, pineapple, passion fruit and tomato juice. These works are mainly focused on the test of different types of membranes and the effect of operating conditions on the permeate flux and retention of juice compounds (Bowden and Isaacs, 1989).

It has been found that polyamide has greater retention of flavors and other constituent's as well higher fluxes than cellulose acetate membrane (Fukutani and Ogawa, 1983). Recovery of apple juice volatiles during RO concentration to 20° Brix was found to be about 80% when using a high resistance (HR) membrane compared a commercial spiral wound cellulose acetate membrane and polyamide membranes (Chua et al., 1987). Different membrane configuration may also affect the retention of flavor compounds during apple juice concentration. The plate and frame configuration had 11.5% permeation of n- hexanal compared with 0.5% for the spiral wound. This was probably due to greater membrane packing and membrane area of the spiral wound (3.90 m<sup>2</sup> of total membrane surface area). The spiral wound system gave lower concentration of n-hexanal in the permeate because of increased rejection and/or of increased flavor compound absorption due to greater membrane surface. (Braddock et al., 1988) reported that volatile compounds except for methanol, ethanol and traces of limonin, were not detected in measurable quantities in the permeate , during the concentration of orange, grapefruit and lemon juices in the range 22-25° Brix at pulp contents of 7-10%, using a composite tubular RO

membrane. At higher temperature, the membrane permeability coefficient is higher, the diffusivity in the solution increases and the viscosity coefficient decreases.

A recent development in RO membranes is the thin- film composite (TFC) membranes, which are made up of polyamide. These membranes consist of a very thin barrier layer on top of a more porous membrane supporting layer usually polysulphone or polyethylene. These membranes give better performance with regard to temperature and pH stability and cleanability, but have almost zero chlorine resistance. Polyamide membranes are also known to have a longer useful life than cellulose acetate membranes (Shew and Willey, 1984).

### 3.2 Microfiltration:-

Microfiltration (MF) is the filtration through an MF- membrane, i.e. a membrane of a pore size of 0.08  $\mu\text{m}$  to 2.5  $\mu\text{m}$  corresponding to a molecular cutoff value 150,000 to 5000,000. Micro filtration (MF) is the process of removing particles or biological entities in the 0.025  $\mu\text{m}$  to 10.0 $\mu\text{m}$  range from fluids by passage through a micro porous medium such as a membrane filter. Although micron-sized particles can be removed by use of non-membrane or depth materials such as those found in fibrous media, only a membrane filter having a precisely defined pore size can ensure quantitative retention. Membrane filters can be used for final filtration or prefiltration, whereas a depth filter is generally used in clarifying applications where quantitative retention is not required or as a prefilter to prolong the life of a downstream membrane. Membrane and depth filters offer certain advantages and limitations. They can complement each other when used together in a microfiltration process system or fabricated device. The retention boundary defined by a membrane filter can also be used as an analytical tool to validate the integrity and efficiency of a system. For example, in addition to clarifying or sterilizing filtration,

fluids containing bacteria can be filtered to trap the microorganisms on the membrane surface for subsequent culture and analysis. Microfiltration can also be used in sample preparation to where intact cells and some cell debris removal are required. Membrane pore size cut-offs used for these types of separation are typically in the range of 0.05  $\mu\text{m}$  to 1.0  $\mu\text{m}$  (Bowen et al., 1995).

Microfiltration is a pressure driven, microporous membrane process used to retain matter as low as 0.2  $\mu\text{m}$  size, but more commonly of 0.1-10  $\mu\text{m}$  size. The matter may include large colloids, small and solid particulates, blood cells, yeast, bacteria and soluble macromolecules. Membrane structure for MF include screen filters that collect retained matter on the surface and depth filters that trap particles at constrictions within the membranes (Zeman and Zydney, 1996).

Microfiltration membranes are available in natural and synthetic polymers like Polypropylene, polycarbonate, polysulphone, polyvinyl chloride, cellulose esters and inorganic materials like alumina, zirconia /carbon composites, carbon / carbon composites, stainless steel and silica. The ceramic membranes have increasingly found application, particularly in biotechnology industry (Cheryan, 1998). The main problem in the practical application of juice is the reduction of permeate flux with time due to membrane fouling, caused by the accumulation of the feed on the membrane surface and adsorption inside the pores cause pore blocking (Bai and Leow, 2002).

### **3.4 Ultrafiltration:-**

A process for concentrating vegetable or fruit juice, in which a permeate stream and a retentate stream are formed by subjecting the juice to an Ultrafiltration step across a hydrophilic membrane having a molecular weight cut off of maximally 80 kDa to a concentration factor of at

least 5, after which the permeate stream is subjected to evaporative concentration. For hydrophilic ultrafiltration membrane there is a great preference for polysulfone and /or polyether sulfone membranes, other suitable hydrophilic membranes are based on cellulose esters, polycarbonate, polyamide, polyether amide and polyether ether ketone. (Westhoff et al., 2010). The retentate of the ultrafiltration step contain the aroma and flavor substance typically semivolatile substance; alpha-pines, limonin, protin and other macromolecules. The permeate comprises amino acids, sugars and a variety of water soluble components.

Ultrafiltration (UF) is the process of separating extremely small particles and dissolved molecules from fluids. The primary basis for separation is molecular size, although in all filtration applications, the permeability of a filter medium can be affected by the chemical, molecular or electrostatic properties of the sample. Ultra filtration can only separate molecules which differ by at least an order of magnitude in size. Molecules of similar size cannot be separated by ultra filtration. Materials ranging in size from 1K to 1000K molecular weight (MW) are retained by certain ultrafiltration membranes. Colloidal and particulate matter can also be retained. Ultrafiltration membranes can be used both to purify material passing through the filter and also to collect material retained by the filter. Materials significantly smaller than the pore size rating pass through the filter and can be dehydrogenated, clarified and separated from high molecular weight contaminants. Materials larger than the pore size rating are retained by the filter and can be concentrated or separated from low molecular weight contaminants. Ultrafiltration is typically used to separate proteins from buffer components for buffer exchange, desalting, or concentration. Ultrafilters are also ideal for removal or exchange of sugars, non-aqueous solvents, the separation of free from protein-bound ligands, the removal of materials of



low molecular weight, or the rapid change of ionic and/or pH environment (Kilduff and Robeson, 1992).

It retains particle of submicron size by ultramicroporous membrane. Typically, UF retains the solute in the 300-500,000 molecular weight range, including biomolecule, polymers, sugar and colloidal particles, lipids, proteins and colloids while small solutes, for example, vitamins, salt and sugars flow through the membrane along with water. Therefore, the possibility of microbial contamination in the permeate stream is minimized. Consequently any thermal treatment and loss of volatile aroma substances are avoided (Menhaj, 1998).

UF is becoming a more attractive unit operation for the clarification of juice compared to the other conventional methods e.g. use of fining agents, diatomaceous earth etc. treatment of various juice such as pear, orange & lemon (Dasgupta et al., 2003).

### **3.5 Diafiltration:-**

Filtration through a membrane, especially a UF membrane, where water has been added to the feed dispersion. It is possible to reduce the amount of permeable solids in the retentate by using Diafiltration (Saugmann et al., 1997). Citrus pulp products are provided which incorporate components from pulp material from a citrus juice source. Naringin, limonin or other bitterant levels are reduced substantially in the pulp components. In particular applications, the debittered citrus pulp is a grapefruit originating bland clouding agent. This separates a citrus juice source into a permeate liquid and a retentate containing virtually all of the pulp present in the citrus juice source. This pulp retentate is subjected to Diafiltration, which reduced levels of bitterents such as naringin and limonin within the pulpy material, and the diafiltration retentate is

processed as or into useful pulp products and /or clouding agents which have blandness characteristics as desired (Chu et al., 2003).

### 3.6 Osmotic Distillation:-

Very recently, osmotic distillation (OD) and membrane distillation (MD) have been proposed as attractive membrane processes allowing very high concentrations (above 65°Brix) to be reached under atmospheric pressure and temperatures near at ambient temperature. In both processes, fruit juice is separated from a receiving phase by a hydrophobic microporous membrane to prevent penetration of aqueous solution, creating air gaps within the membrane. The driving force of the process is given by a water vapor pressure gradient across the membrane, causing water vapor transfer across the pores from high-vapor pressure side to the low one. In MD process, water vapor pressure difference is generated by the temperature difference in the two sides of the membrane. On the other hand, in OD process, the difference in water activity between the juice and a hypertonic salt solution (concentrated brine stripper) induces a water vapor pressure gradient across the membrane at room temperature. Since the driving force is not a hydraulic pressure difference, very high concentrations compared to RO can be achieved by both MD and OD processes. In this case, osmotic solution is cooled and the feed solution is slightly heated in order to enhance water flux (Cassano and Drioli, 2007).

As a pretreatment of concentration, clarification is necessary in order to obtain a bright, clear product with low viscosity which makes concentration easier with less fouling and greater concentration. Clarification is generally conducted by using fining agents as well as by using membranes. The common fining agents used for clarification of fruit juices include bentonite, gelatin or a combination of these compounds. Bentonite is effective for protein stabilization.

However, a very little effect of bentonite on polyphenol removal has also been reported. Gelatin is a positively charged molecule in the low pH range of fruit juices and reacts with negatively charged phenolics. On the other hand, clarification based on membrane processes, particularly ultrafiltration (UF) and microfiltration (MF), have been replacing conventional fining for clarifying fruit juices with the advantages of elimination of fining agents together with their associated problems and production with a continuous simplified process.

Osmotic distillation is a recent membrane process, also known as osmotic evaporation, membrane evaporation, isothermal evaporation and gas membrane extraction (Deblay, 1995) which has been successfully applied to the concentration of liquid foods such as milk, fruit and vegetable juice, instant coffee and tea and various non food aqueous solutions. These techniques can be used to extract selectively the water from aqueous solutions under atmospheric pressure and at room temperature, thus avoiding thermal degradation of the solutions (Ben and Aim, 1996). It is therefore particularly adapted to the concentration of heat sensitive product like fruit juices.

Sheng et al., 1991 studied the effect of operating conditions (juice flow rate, juice concentration and temperature) on the OD flux during the concentration of apple, orange and grape juice through a PTFE membrane with pore size of 0.2  $\mu\text{m}$  and an overall thickness of 100  $\mu\text{m}$  and they were observed the OD flux decreased with the increase of juice concentration and it depends strongly on the osmotic pressure difference between the aqueous streams.

Membrane distillation is a relatively new membrane process in which two aqueous solutions, at different temperatures, are separated by a microporous hydrophobic membrane (Calabro et al., 1994). In this condition a net pure water flux from the warm side to the cold side

occurs. The process takes place at atmospheric pressure and at temperature that may be much lower than the boiling point of the solutions. The driving force is the vapour pressure difference between the two solution- membrane interfaces due to the existing temperature gradient. The phenomena can be described as a three phase sequence: (1) formation of a vapour gap at the warm solution-membrane interface; (2) transport of the vapour phase through the microporous system; (3) its condensation at the cold side membrane-solution interface (Molinari et al., 1992).

The most suitable material for MD membranes includes polyvinylidene fluoride (PVDF), polytetrafluoroethylene (PTFE) and polypropylene (PP). The size of micropores can range between 0.2 and 1.0  $\mu\text{m}$ . the porosity of the membrane will range from 60% to 80% of the volume and the overall thickness from 80-250  $\mu\text{m}$ , depending on the absence or presence of support. In general, the thinner the membrane, the greater the flux rate. The membrane configuration used includes flat sheet, spiral wound and hollow fiber. Because the MD can be carried out at atmospheric pressure and at a temperature which can be much lower than the boiling point of the solution, it can be used to concentrate solutes sensitive to high temperature (e.g. fruit juice), also at high osmotic pressure. Therefore MD has received great attention as techniques for concentrating fruit juices (Durand and Dornier, 1996).

UF is a powerful Method for removing natural polymers (polysaccharides, proteins) from fruit and vegetable juice. So far, studies on concentration of fruit juices by OD and MD processes have mainly focused on enhancing water flux, hence the effectiveness of the process by integration of other membrane processes, namely ultrafiltration, microfiltration and reverse osmosis. (Onsekizoglu et al., 2010).

#### **MEMBRANE CLEANING METHODS:-**

The membrane can be polluted and blocked up because the separation substance and impurity can deposit on the surface of the membrane. So the system must contain the process of cleaning membrane. There are several different membrane cleaning methods, such as forward flush, backward flush and air flush. The effective cleaning is the important ways and means to extend the using life .the cleaning methods include chemical cleaning and physical cleaning (Karode et al., 2001).

- 1) Forward flush: When forward flush is applied, membranes are flushed with feed water or permeate forward. The feed water or permeate flows through the system more rapidly than during the production phase. Because of the more rapid flow and the resulting turbulence, particles that are absorbed to the membrane are released and discharged. The particles that are absorbed to membrane pores are not released. These particles can only be removed through backward flushing (Karode et al., 2001). When forward flush is applied in a membrane, the barrier that is responsible for dead-end management is opened. At the same time the membrane is temporarily performing cross-flow filtration, without the production of permeate. The purpose of a forward flush is the removal of contaminants on the membrane through the creation of turbulence. A high hydraulic pressure is in order during forward flush.
- 2) Backward flush: Backward flush is a reversed filtration process. Permeate is flushed through the feed water side of the system under pressure, applying twice the flux that is used during filtration. When the flux has not restored itself sufficiently after back flushing, a chemical cleaning process can be applied (Lefebvre et al., 1998) chemical cleaning is a cleaning membrane method of chemical medicine and impuity in the surface

of membrane reacting in each other like Acid-solution cleaning in which Chlorhydric acid, citric acid, oxalic acid can be used as usual agent, the pH value of acid-solution is 2 to 3 cyclic cleaning or cyclic cleaning after soaked 0.5h to 1h can eliminate inorganic impurity effectively. Alkali solution cleaning in which, The main agent is NaOH, the pH value of solution is about 10 to 12, cyclic cleaning or cyclic cleaning after soaked 0.5h to 1h can eliminate impurity and grease effectively and Oxide cleaning agent, in which Cleaning UF membrane with the water solution of 1%-3%  $\text{H}_2\text{O}_2$ , 50-500mg/h NaClO can eliminate faith, bacterium.  $\text{H}_2\text{O}_2$  and NaClO is the common germicide (Mansouri et al., 1999).

- 3) Balanced pressure cleaning method: Close the valve of UF water, open the outlet valve of concentration water and wash the surface of membrane by raising flow speed. It can eliminate most of the swampy impurity on the surface of membrane (Rao et al., 1987).
- 4) Ultra water cleaning method: The solution ability of ultra water is strengthen, the loose filth on the surface of the membrane can be cleaned with UF water first, and then cleaned with cyclic pure water (Schofield et al., 1987).

## COST CONSIDERATION AND ENVIRONMENTAL IMPACT

Membrane processes are considered as very energy efficient compared to many other separation processes. However, the energy requirement of a process is only one cost determining factor. Investment and maintenance related costs contribute often significantly to the overall process costs. Other factors that must be considered are pre- and post-treatment procedure, the required product and especially the composition of the feed mixture which gas to be treated (Cheryan, 1998). For the concentration of apple juice and passion fruit juice micro and

ultrafiltration can be used. In this processes the energy requirement are quite low. However, micro and ultrafiltration are competing with biological treatment or sand bed filtration which need even less energy. In orange juice concentration only economical membrane process is reverse osmosis which is competing with the distillation techniques. As far as energy consumption is concerned, reverse osmosis is the more energy efficient process (Cassano et al., 2011). However, it has to be taken into account that in reverse osmosis the pressure-generating pumps are driven either by electric or combustion engines.

The environmental impact of all membrane processes is relatively low. There are no hazardous chemicals used in the processes that have to be discharged and there is a no heat generation (Hernandez, 1992).

#### **THE FUTURE OF MEMBRANE SCIENCE AND TECHNOLOGY:-**

In many applications today's membranes and processes are quite satisfactory while in other applications there is a definite demand for further improvements of both membranes and process. For orange and apple juice concentration by reverse osmosis, e.g. there are membranes available today that are quite satisfactory as far as flux and salt rejection concerned, and the processes are proven by many years of operating experience ( Labuzza, 1970). In micro- and ultrafiltration the properties of present membranes are satisfactory. However, there are other components such as the process design, process control, application know-how, and long-term operating experience that are of importance in the use of micro and ultrafiltration in the chemical and food industry. Here, concentration polarization and membrane fouling play a dominant role and new membranes modules and process design concepts which provide a better control of membrane fouling resulting in a longer useful life of the membranes are highly desirable. In

other membrane processes such as gas separation, pervaporation, membrane reactors, etc. the situation is quite different. Here, better membranes, improved process designed and extensive application know-how and long-term experience are mandatory to establish membrane processes as a proven and reliable technology (Drioli et al., 1999).

In addition to the established membrane processes and applications, new membrane operations such as membrane contactors and membrane reactors are growing at industrial level and becoming common unit operations in process engineering, contributing also to the overall impact of membrane engineering on any industrial production (Ho et al., 1992). It is also particularly important that all membrane operations are well consistent with the requirements of the process intensification strategy and of a sustainable industrial development.

Gunko et al., 2006 studied the concentration of apple juice by Direct Contact Membrane Distillation, using Vinilydene Fluoride Hydrophobic MF with a nominal mean pore size of  $0.45\mu\text{m}$ . Experiment show that at constant temperature of juice in the hot cell, an increase in the flux permeate of DCMD resulted in reducing the temperature of the cooling water in the cold cell. The deterioration of hydrodynamic condition of the process increase the influence of temperature and concentration polarization, as a result the rate of the mass transfer through the membrane is reduced, leading to a reduction in the permeate flux. This work show that the concentration of apple juice by DCMD must be up to 50% of soluble substance content when the permeate flux reaches about  $91/\text{m}^2\text{h}$ .

Onsekizoglu et al., 2010 studied that clarification of apple juice by combined application of fining agents (gelatin and bentonite) and UF found an effective methodology for reducing haze formation. Especially physical quality attributes like color were improved markedly



following UF through 10 kDa membrane. UF didn't change the composition of apple juice. Only some minor components, mainly phenolics and trans-2-hexenal were retained by 10 kDa membranes. It was also resulted a slight decrease in HMF content in comparison with UF through 100 kDa membrane. Following clarification, membrane distillation, osmotic distillation and coupled operation of osmotic and membrane distillation processes allowed to concentrate the apple juice up to the same TSS content as that obtained with thermal evaporation, at low temperatures. The clarified apple juices with initial TSS contents of 12°Brix were subsequently concentrated up to TSS contents of 65°Brix by membrane-based concentration processes. The new membrane-based concentration techniques were very efficient since the concentrated juice presented nutritional and sensorial quality were very similar to that of the original juice especially regarding the retention of bright natural color and pleasant aroma, which are considerably lost during thermal evaporation.

Furthermore, among all the concentration treatments applied, only thermally evaporated samples resulted formation of HMF, the potential indicator of Maillard reactions. Phenolic compounds, organic acids and sugars were very stable against all concentration processes and also in thermal evaporation. In aroma profile, the comparison of the effects of different concentration processes on aroma compounds retention were studied in terms of trans-2-hexenal, because trans-2-hexenal was the most abundant aroma compound in concentrated apple juices. In thermal evaporation technique it was markedly lost in apple juice concentrates. A higher retention of trans-2-hexenal was observed with the osmotic evaporation process compared to membrane distillation process. The coupled operation of OD and MD may be the best alternative among all the concentration process examined, retaining almost all the trans-2-hexenal content of

the clarified juice because of the considerably low operating temperature and short processing time.

Warczok et al., 2004 studied that the concentration of apple juice by Nanofiltration at low pressure. The aim of this work was to find a proper membrane and the most adequate process condition to reach the highest concentration degree of the juices, pressures between 8 and 12 bar and temperature of 25-30 °C were considered. Pretreatment of apple juice is done with the help of centrifuge to remove the pulp from apple juice at 2000 rpm for 10min. To understand better and to optimize the process, nanofiltration membrane were characterized by using atomic force microscopy (AFM) and scanning electron microscopy (SEM), also an evaluation of irreversible fouling was carried out. The result indicated that in membrane selection both retentation and permeation values should be considered, and that irreversible fouling of fruit is relatively low ( $30 \pm 8\%$ ). The juice solutions can be concentrated by NF and the membrane tested that gives higher concentrations. The decrease in permeate flux is significantly greater in juice solutions than fructose solutions. This is related to the complex composition of juices.

AFM and SEM analysis is a good tool for characterizing the topology (mean pore distribution, roughness and distribution of separation-layer particles) and morphology (thickness of membrane layers). They concluded that roughness does not depend on structure, density and t membranes mostly

Bruijn and Borqueuz, 2006 evaluated the fouling mechanisms of a Carbosep M8 membrane during cross- flow ultrafiltration of apple juice. They developed the new fouling model that simultaneously considers membrane blocking within the pores, at the pore mouths and by cake formation at the membrane surface. They used a pilot – scale cross- flow UF unit

equipped with a tubular Carbosep M8 membrane consisting of a porous carbon support and an active zirconium layer with manufacturer reported MWCO of 50 kDa. When operating UF at a transmembrane pressure of 150 kPa and a cross- flow velocity of 7 m/s, fouling was minimal with a gradual decrease of the relative contribution of cake formation; however, transmembrane pressure still exceeds critical pressure and the result obtained that the fouling model predicts no cake formation at a cross- flow velocity of 7.4 m/s and a transmembrane pressure of 150 kPa or at a cross- flow velocity of 7.0 m/s and a transmembrane pressure of 120 kPa. Under these conditions, internal membrane blocking would be the only mechanism responsible for the decrease of permeates flux.

Tajchakavit and Boye, 2001 studied the Post-bottling haze formation in clarified apple juice produced under a variety of processing condition. Turbidity of the juice was monitored at intervals during a 6-month of storage period. The clear juice obtained was ultrafiltered through 100 kDa MWCO ultrafiltration membrane at 50°C. The clarified juices was then subjected to final pasteurization at 95°C for at least 30 s and packaged in sterilized glass containers. Changing the pasteurization temperature of raw juice and omitting the addition of amyloglucosidase prevent haze formation.

Omission of the fining procedure, however, resulted in an increase in turbidity of 1.36 NTU and visual observation of larger quantities of haze. Fining with a gelatin/ bentonite ratio 1:1 gave least haze. Thermocouglution treatment applied to the juice prior to UF also did not retard or prevent haze formation. UF at lower processing temperatures resulted in the development of less haze. They observed an increase in turbidity of over 14 NTU at high temp and the result

suggest that optimization of the fining procedure and UF could be used to retard post- bottling haze in apple juice.

Lukanin et al., 2003 investigated the potential for membrane distillation (MD) flux treatment in apple juice concentration by removal of biopolymers. Introduction of additional enzymatic deproteinization to conventional treatment with pectinase / amylase complexes and application UF as a clarification method that provide an obtaining of clarified apple juice with minimal content of biopolymers. They used the Polysulphonamide commercial membrane with a MWCO of 50,000Da in order to avoid the permeation of enzymes, whose molecular weights are in the range of 40,000 -100,000 kDa. Such a pre- treatment enhances trans-membrane flux during concentration of clarified apple juice by MD.

This has been attributed to a reduction in juice viscosity. Lower viscosity improved hydrodynamic condition in membrane channel thereby decreasing concentration and temperature polarization. With increasing juice concentration, difference in process productivity in MD becomes more notable for apple juice with different initial viscosity.

Alvarez and Riera, 1998 evaluate the UF performance to clarify apple juice by using a Carbosep membrane of 15 and 150 kDa, tangential velocity of 2 and 7 m/s and transmembrane pressure of 150 and 400 kPa. They studied the effect of operating condition on membrane fouling, process efficiency and juice quality. Averages permeate flux varied between 56 and 157 L/ (m<sup>2</sup>h). Color, clarity and turbidity of juice improved significantly after UF. They observed that fouling resistance increased with high transmembrane pressure and low feed velocity. At high pressure, a high flow rate decreased the rate of fouling, improving average permeate flux, but increasing energy consumption.

Sarkar et al., 2008 evaluate that the effect of external electric field on the enhancement of permeate flux during clarification of mosambi juice by ultrafiltration. This investigation has been carried out using a 50 kDa molecular weight cut- off flat sheet polyethersulfone membrane in cross- flow ultrafiltration under laminar flow conditions for a wide range of operating conditions. This study highlights the potential utility of using external D.C. electric field to increase the productivity of the process. For example, at a cross-flow velocity of 0.12 m/s and a transmembrane pressure of 360 kPa the permeate flux increase from 8.65 to 11.75 L/m<sup>2</sup> h by applying electric field of 400 v/m, and the additional electric power consumption is 1.16 kwh/m<sup>3</sup> of permeate. They observed the gel concentration is about 48.5 kg/m<sup>3</sup> and the effective diffusivity of the solute in the juice is about  $6 \times 10^{-11}$  m<sup>2</sup>/s. the effective viscosity in the concentration boundary layer adjacent to gel layer is  $7.03 \times 10^{-3}$  Pa s which is about 7 times the bulk viscosity.

Rai et al., 2007 analyzed the permeate flux and quality using various molecular weight cut off membranes of depectinized mosambi juice at fixed pressure and stirring speed. Pectinase from aspergillus nigar was used for the pretreatment of mosambi is used. The flux decline during filtration were quantified, this analysis show that the cake filtration is the main reason for flux decline. The complete and partial pore blocking mechanism may prevail within first few minutes from the start of filtration.

They used UF membrane were thin film composite Polyamide of MWCO 10k, 20k, 30k, 50k and 100k and MF of Cellulose Acetate of 0.2µm. enzymatic treatment of time 100 min, temp 42°C and enzyme conc. (0.0004% w/v), and observed that both the permeate flux and permeate quality are not affected by much selection of higher MWCO membrane, selection of higher

MWCO membranes leads to irreversible fouling, which cause reduction of permeate flux and finally the life of the membrane. Therefore, a membrane up to 50k is suitable for the clarification of mosambi juice.

Nandi et al., 2009 studied the MF of mosambi juice using low cost ceramic membrane prepared from locally available inorganic precursors. Characterization of the prepared membrane was done by SEM analysis, porosity determination and pure water permeation experiments. The average pore diameter, total porosity and hydraulic resistance of the membrane were evaluated as 0.285 $\mu$ m, 23.6% and 9.26 $\times 10^1$  respectively. They performed the Dead-end MF for centrifuged mosambi juice [CJ] and enzyme treated centrifuged mosambi juice [ETCJ]. They observed that after MF, important properties like TSS, pH, acidity and density of both CJ and ETCJ were almost unaffected, significant improvement in juice colour, clarity and AIS was observed. They also observed that the clarified juice can be stored in refrigerated condition for more than 30 days without significant change in juice quality. They used the different membrane pore blocking models to analyze the observed permeate flux decline.

Rai et al., 2004 used the Neural network models to describe the permeate flux and permeate concentration during the UF of synthetic fruit juice and mosambi juice. It aims to predict the permeate flux and the processing time. They used a multi-layer feed forward network structure with input, output and hidden layer and a thin film composite polyamide membrane of MWCO 50,000 kDa. The permeability of the membrane was found to be  $1 \times 10^{-10}$  m/ Pa using distilled water.

Two neural network models are constructed to predict the permeate flux and the total soluble solids in the permeate using the filtration data of the synthetic fruit juice and mosambi

juice. The modeling result showed that there is an agreement between the experimental data and predicted values, with mean absolute errors less than 1% of the experimental data and the trained networks are able to capture accurately the non- linear dynamics of synthetic fruit juice and the actual mosambi juice even for a new condition that has been used in the training process.

Rai et al., 2005 studied the characteristics of synthetic sucrose, pectin mixture and enzymatically treated mosambi juice in a stirred continuous cell, this model is based on Gel layer theory They used a 50,000 MWCO of thin film composite polyamide membrane of UF and observed that the gel layer is in compressible at higher operating pressure in case of mosambi juice and the nutritional value of the original juice are retained by the clarified juice.

Dasgupta et al., 2003 investigate the mosambi juice treated with pectinase at different durations (40-141 min), temperature (32-49 °c), and at various concentration level of enzyme (0.0004- 0.0014 w/v %). The effect of the enzyme treatment condition on various parameters like apparent viscosity, clarity and alcohol insoluble solid were studied. The optimum process condition, were determined employing a second- order central composite rotatable design in combination with response surface methodology. Result showed that apparent viscosity and alcohol insoluble solid decreased with temperature and enzyme concentration and clarity of the juice increase with temperature and the enzyme concentration.

Cassano and Drioli, 2007 evaluated the O.D. process for concentrating the clarified kiwifruit juice, taking into account the impact on the product quality in terms of ascorbic acid content and total antioxidant activity (TAA) and also the performance of O.D. process in terms of flux and concentration factor.

They purchased the fruit from local market and were manually washed with running water in order to remove foreign material from the skin. Hence they were manually cut up and then pulped using a multiple shaker liquidizer in order to facilitate and accelerate the action of pectolytic enzymes. The clarified kiwifruit juice containing TSS of 9.4°Brix was concentrated to 66.6°Brix using a bench plant equipped with a liquid-cell with polypropylene hollow fiber membrane and observed that the O.D. process has no influence on the acidity but reduction of 87% of vitamin C was observed in the clarified juice and also TAA of the clarified juice maintained during the process.

An integrated membrane process for the production of concentration kiwifruit juice and its aroma recovery designed by (Sindona et al., 2006). Fresh depectinised kiwifruit juice was clarified by the ultrafiltration process then concentrated by osmotic distillation, as an alternative to the traditional vacuum evaporation, up to total soluble solids (TSS) content higher than 60°Brix at 25°C. Purchased the fruit from local market and were manually washed with running water in order to remove foreign material from the skin. Hence they were manually cut up and then pulped using a multiple shaker liquidizer. For this purpose they used a tubular membrane module of polyvinylidene fluoride, 15 kDa. Pervaporation runs were carried out before and after each membrane unit operation in order to identify the best configuration giving the minimal loss of aroma compounds. For the majority of aroma compounds detected, they observed that the enrichment factor in the permeate of the fresh juice was higher than the clarified and concentrated juices. This result show that the use of PV for the removal and enrichment of aroma compounds directly from the fresh juice, before any concentration process. It has different



advantage in terms of: reduction of clarification times, simplification of the clarification process and possibility of operating at room temperature for preserving the freshness, aroma and nutritional value of the juice, improvement of the quality of the final product and of the production processes. Suspended solids in fresh kiwifruit juice were completely removed by UF and the resulting clarified juice had lower viscosity and negligible turbidity. Only a slight decrease of the TAA and of the ascorbic acid was observed in the concentrated juice with respect to the fresh juice.

The effect of high pressure and heat treatments on peroxidase (POD) activity in kiwifruit juice has been investigated by (Liang et al., 2008). Kiwifruits were cleaned with detergent and rinsed twice with distilled water. Then, they were peeled with a surgical blade to prevent damage of the outer pericarp tissue and cut into halves along their major axis. Peeled kiwifruit were suspended in 200 ml of 50 mM sodium phosphate buffer, with 1 M NaCl, mixed for 15 s in a waring commercial blender. The resulting homogenate was centrifuged for 20 min in a centrifuge and the supernatant was filtered through four layers of cheese cloth. Pressure level ranging from 200 to 600 MPa and temperature varying from 10 to 50 °C were applied for up to 30 min. Assays were carried out on crude peroxidase in kiwifruit juice and on partially purified peroxidase in a model system. Pressure higher than 400MPa could be combined with mild heat ( $\leq 50^{\circ}\text{C}$ ) to accelerate enzyme inactivation. They observed that prolongation of the exposure time had no great effect after the first 15 min. and the slope of POD in a kiwifruit juice at 30°C was slightly decreased compared with that in a model system. Furthermore, the optimum pH was obtained as 6.0-8.5. There are several POD isoenzymes in kiwifruit and that resistance to pressure is responsible for the activity still remaining after treatment.

Cassano et al., 2007 performed an experiment according to the recycle mode the effect of TMP, axial flow rate and temperature on permeate flux by using tubular membrane of PVDF 15 kDa. The result of this experiment showed that the flux increased with temperature from 20 to 30 °C and with axial feed flow rate from 300 to 700 l/h and flux-pressure curves showed no increase in permeates fluxes for TMP values higher than 90kPa. i.e. if maximum permeation flux, minimum fouling and quality of juice are the requirement, the best condition should be at 25°C of temp, 90 kPa of pressure and 700 l/h of flow rate.

Clarified kiwifruit juice has been produced in experimental tests carried out according to the batch concentration mode working in optimal operating and fluid dynamic conditions. For a pretreatment purchased the fruit from local market and were manually washed with running water in order to remove foreign material from the skin. Hence they were manually cut up and then pulped using a multiple shaker liquidizer in order to facilitate and accelerate the action of pectolytic enzymes. After pulping sodium sulphite was added in order to inhibit the enzyme polyphenol oxidase that determines a browning of the pulp. The quality of juice analyzed in terms of TAA, content of ascorbic acid, suspended solid, turbidity and viscosity. The UF process permeated a good level of clarification reducing totally the suspended solids and the turbidity of the fresh juice. In the permeate a 16% reduction of ascorbic acid was observed with respect to the fresh juice and reduction of the TAA was lower than 8%.

Cake layer and irreversible fouling gave a minimum contribution to the total resistance (2.23% & 2.75%) while the contribution of the reversible fouling was more significant (24.4%). A good restore of the hydraulic permeability of the membrane was observed after a cleaning treatment performed by using alkaline and acid detergents. Thus, the flux decline during UF

could be ascribed to fouling layers formed by a combination of suspended particles and adsorbed macromolecular impurities.

Jiao et al., 2004 described the research efforts to develop and optimize an integrated membrane process on laboratory scale, for the production of concentrated kiwifruit juice as alternative to the traditional vacuum evaporation. The concentration of fruit juice is industrially performed in order to reduce storage, packaging, handling and shipping costs. Fresh depectinated juice clarified by UF process. The experimental tests were performed according to the total recycle mode the effect of TMP, axial flow-rate & temperature on permeate flux. Clarified juice was produced in experimental tests to the batch concentration mode working in optimal operating and fluid dynamic conditions. For a pretreatment purchased the fruit from local market and were manually washed with running water in order to remove foreign material from the skin. Hence they were manually cut up and then pulped using a multiple shaker liquidizer in order to facilitate and accelerate the action of pectolytic enzymes. The OD process was used to concentrate the juice up to TSS content higher than 60°Brix at 25°C. The effect of various operating parameters on vapours flux was studied. An average evaporation flux of almost 1kg/m<sup>2</sup> was obtained using calcium chlorides dehydrate at 60 w/w% as a brine solution.

The UF process permitted a good level of clarification reducing the suspended solid and the turbidity of the fresh juice. Additional advantages are in terms of: increasing of yield in terms volumes of clarified juice produced, possibility to avoid the use of gelatins, adsorbents and other filtration coadiuvant. A little reduction of the total antioxidant activity was measured during the UF-OD treatment where the ascorbic acid values in the samples coming from the membrane process and their contribution to the TAA were constant.

Conidi et al., 2008 study the influence of the UF on the composition of some bioactive compounds of the kiwifruit juice in order to develop a natural product which can be used to fortify foods and beverages and at the same time the effect of TMP and temperature on the permeate flux also investigated in order to identify the optimal operating conditions for the processing of the juice. An optimal TMP values occurred at 0.6-0.65 bar in different conditions of cross flow velocities. For this purpose they were used a UF membrane of Cellulose Acetate with MWCO 30 kDa. The kiwifruit juice was clarified in optimal operating conditions, according to batch concentration mode, up to a final volume reduction factor (VRF) of 2.76.

The analysis of flux decay according to fouling models reported that the formation of a cake layer covering the entire surface of the membrane is the main cause of the membrane fouling. Most bioactive compounds of the depectinised kiwifruit juice recovered in the clarified fraction of the UF process. They observed that the recovery of glutamic, folic, ascorbic acid and citric acid in the clarified juice with respect to the initial feed was dependent on the final VRF of the UF process: an increase of the VRF determines an increase of these compounds in the clarified juice. The rejection of the UF membrane towards these compounds in the range 0-4.3%.

The UF process: an increase of the VRF determines an increase of these compounds in the clarified juice. The rejection of the UF membrane towards these compounds in the range 0-4.3%.

Jesus and Leite, 2007 evaluated the orange juice concentrated by RO in a plate and frame pilot plant with 0.72 m<sup>2</sup> of filtration area composed polysulphone/polyethylene composite layer membranes. They evaluated three TMP 20, 40, & 60 bars and obtained the concentration factor 2.3, 3.8 & 5.8 at this pressure, the final soluble solid contents of 16, 28 & 36°Brix. The vitamin C

content increased from 29.3 mg ascorbic acid /100g to 53.9, 82.7 & 101.1 mg/100g. The sensory evaluation of the reconstituted juice obtained by dilution of the concentration juice and observed that these products lost its characteristics aroma and flavor when compared to the fresh orange juice. In second experiment, some sensory attributes were evaluated in the reconstituted juice obtained by two concentration process i.e. RO and thermal evaporation. Result of this process showed that the juice from RO process had a best preserved flavor so they conclude that to concentrate the orange juice by RO process maintaining its sensory and nutritional characteristics.

Cassano et al., 2007 studied the orange juice clarified by cross-flow UF by using tubular Polyvinylidene Fluoride membrane. They performed the experiment according to the total recycle mode the effect of TMP, axial feed-flow rate and temperature on permeate flux. The clarified juices were carried out according to batch concentration mode working in optimal operating and fluid dynamic conditions. The fouling mechanisms during cross-flow UF were identified by estimation of the model parameters according to a non-linear regression optimization procedure.

Analysis of this result showed that, in the fixed operating conditions of TMP and temperature the fouling mechanism evolves from a partial to a complete pore blocking condition in dependence of the axial velocity. The quality of the sample obtained from UF process evaluated in terms of TSS, suspended solid, TAA, ascorbic acid and flavor & obtaining the clarified juice were highly similar to the fresh juice expect for insoluble solid which were concentrated in the retentate stream.

Sanchez et al., 2010 study the cryoconcentration of orange juice using a pilot plant for cryoconcentration of liquids using a cold surface. The orange juice used in this study (200L) were pasteurized and clarified juice with a concentration of 11.1° Brix. The evaluation in time of the concentration of solid in the juice & in the ice was analyzed during the process. They found that the concentration of solid in juice showed a linear increase in time at a rate of 0.75°Brix.

Gianni et al., 2008 evaluated the production of high quality concentrated blood orange juice according to by a new integrated membrane process, alternative to thermal evaporation in terms of the total antioxidant activity and of the bioactive antioxidant components of the juice like ascorbic acid, anthocyanins, hydroxycinnamic acid and flavanones etc. the process were based on the initial clarification of freshly squeezed juice by UF by using a tubular module of PVDF membrane with MWCO 15 kDa. Blood orange juice were produced from fruit ultivated, TSS concentration of the raw juice was about 12.0-12.6°Brix with a pH =3.5. Concentrated orange juice was produced by a multiple effectthermally accelerated short time evaporator at a final concentration of 56.3°Brix. The clarified juice was successively concentrated by two consecutive processes, first RO used as a pre-concentration technique up to 25-30°Brix, then OD up to a final concentration of about 60°Brix. During the concentration process of the liquid fractions, a slight decrease of total antioxidant activity were observed -15%, which was due to the partial degradation of ascorbic acid (ca.-15%) and anthocyanins (ca, -20%). Nevertheless, this degradation was lower than that observed with thermally concentrated juice i.e. TAA,-26% ascorbic acid, -30% anthocyanins, -36%. The possibility to operate at room temperature allowed reduction in thermal damage and energy consumption. On the basis of this results, the integrated membrane process may be proposed as a valuable alternative to obtain high quality concentrated

juice, as the final product showed a very high antioxidant activity and a very high amount of natural bioactive components, showing a brilliant red colour and a pleasant aroma, characteristics that were significantly lost during traditional thermal evaporation.

Mayor et al., 2011 studied the reverse osmosis concentration of press liquid from orange juice solid waste. An alternative for these wastes is their pressing with lime to obtain a press liquor stream and a dried solid for cattle feeding. They performed the preconcentration of two types of synthetic press liquor (with and without pectin) and assessed the fouling mechanism as well as membrane resistance to the permeate pass and also evaluated the evolution of the concentration process in terms of some selected parameters like COD, TSS, TDS and the effect of pectin on the press liquor concentration.

The RO membrane of Aromatic polyamide was used. It was found that, regarding the feed solution with pectin, WP, it was only possible to perform the experiment at 50 bar of applied pressure to reach a VRF value up to 1.2 due to high viscosity of the solution that made difficult its flow through the RO system and in WOP experiment, there is dependence of the initial permeate flux with the TMP. Moreover, there is a clear flux decline in all cases, and the higher is TMP, the higher is the slope of flux decline at early stages of concentration and it is also observed that necessary time to achieve the 2.1 VRF for the WOP solution decreases dramatically with the applied pressure (7320, 1980 and 780 s for TMP 20, 35 and 50 bar).

They also showed that at TMP 20 bar with the feed solution WOP, it was possible to distinguish two different flux decline mechanism identified as the dominant fouling mechanism during later stages of concentration times, the dominant fouling mechanism was the complete pore blocking. Independently of the fouling mechanism that describes the flux decline, the

membrane fouling leads to an increment of the total resistance of the membrane that reduce the capability of obtaining permeate flux. The increment of resistance is due those components of the feed solution that remain on the imperfections and on the membrane surface and lead to a decay of the water permeability of the membrane.

Laorko et al., 2010 investigated the membrane property on permeate flux, membrane fouling and quality of clarified pineapple juice. Fresh pineapples were cleaned by tap water after the shells were peeled by a stainless steel knife, the fresh pineapples were chopped into pieces and the juices were extracted by a hydraulic press. Before using the separation, the pineapple juice was treated with 0.03% of commercial pectinase at room temperature. Both MF (membrane pore size of 0.1 and 0.2  $\mu\text{m}$ ) and UF (MWCO of 30 and 100 kDa) membrane were employed. They observed that the MF membrane with pore size of 0.2  $\mu\text{m}$  gave the highest recovery of phytochemical compounds including vitamin C (94.3%), total phenolic content (93.4%) and DPPH free radical scavenging capacity (99.6%). The membrane pore size and MWCO did not affect the oxygen radical absorbance capacity. Total variable count, yeast, mold and coliforms were removed completely by all membranes employed. The result indicate that membrane filtration with a pore size of 0.2  $\mu\text{m}$  could serve as a cold sterilization process which could achieve the preservative of phytochemical compounds this gives perfect clarification and sterilization in one step.

The permeate flux during filtration of pineapple juice with total recycle mode is studied, in general the flux rapidly declined at the initial stage of the process. Afterwards, the smother and the slower decline towards a quasi steady tended in turn, to be attributed to fouling due to pore blocking and cake built up. The highest permeate flux was obtained from a 0.2  $\mu\text{m}$



membrane while the highest irreversible fouling ( $9.73 \times 10^{12} \text{ m}^{-1}$ ) was obtained from this membrane as well. However there was no difference in permeate flux between 0.2 and 0.1  $\mu\text{m}$  membrane. The lowest irreversible fouling ( $1.58 \times 10^{12} \text{ m}^{-1}$ ) was observed from 0.1  $\mu\text{m}$  membrane. According to the highest permeate flux, total phenolic content and antioxidant capacity, a membrane with a pore size of 0.2  $\mu\text{m}$  was considered to be the most suitable membrane and the cross-flow velocity and TMP did not have significant effect on the phytochemical properties of clarified pineapple juice.

Carvalho et al., 2008 aimed to evaluate the loss of sugars (glucose, fructose and sucrose) in pineapple juice, hydrolyzed with commercial pectinase (Ultrasym 100G) alone and combined with a cellulose (Celluclast) as a pre-treatment, and after clarification by cross flow micro- and ultra-filtration, using two different module geometries (plate/frame and tubular systems) to select the membrane process that would best preserve these nutrients. Membranes of polysulfone (PS), polyethersulfone (PES) and polyvinylidene fluoride (PVDF) to micro- and ultra-filtration were used. The membrane pore diameters and cut-offs were: 0.1, 0.45  $\mu\text{m}$ , and 50, 100 kDa (PS), and 0.3  $\mu\text{m}$  and 30–80 kDa (PES and PVDF). All processes were operated at different trans-membrane pressures (TMP), at room temperature ( $25^\circ\text{C} \pm 2^\circ\text{C}$ ). The sugar contents of the clarified pineapple juices determined by HPLC revealed significant differences at a 5% level. These results showed that the membrane pore diameters or cut-offs as well as the module geometry influenced the clarified juice sugar contents. It was observed that the sugar content was more reduced when the 30–80 kDa tubular membrane at 1.5 bar was used for pineapple juice clarification. Although the best total sugar recoveries had been observed in juices clarified with

polysulfone membranes (50 kDa – 7.5 bar), the use of 0.3 lm PES was more attractive and appropriate due to its tubular configuration and module geometry.

Sangsuwan et al., 2008 experimented on fresh-cut pineapple with different films and their effects on microbial control and fruit quality during storage at 10°C. For the pretreatment of the fruit the TSS of the pineapple juice in the range of 17.0-19.4%. Whole fruit were washed with 500 mg/l chlorine solution. The blossom and stem ends were discarded. All knives, cutting boards and other equipment which come into contact with the fruit were sanitized by immersing in 1000mg/l chlorine solution for 30 min before cutting. Three types of films were used in this experiment: a commercial stretch film, an experimental chitson/methyl cellulose film and a chitson/methyl cellulose film incorporating vanillin as a natural antimicrobial agent. They observed that the chitson/methyl cellulose film rapidly reduce the number of *Saccharomyces cerevisiae* yeast inoculated on pineapple. Vanillin film was more efficient than chitson/methyl cellulose in reducing the number of yeast, which decreased by 4 logs in fresh cut pineapple on 6 days. Vanillin film increased the intensity of yellow color of pineapple. Pineapple removed from stretch film had higher respiration rates and ethanol contents than other treatments.

Jiratananon and Chanachai, 1996 investigated the effect of operating variables on permeation flux and resistance for the UF of passion fruit juice. 0.01ppm pectinase was added to the hydrolyse pectic substance. The juice was then subjected to a centrifugal action at 14000×g to remove starch and the pasteurization process was conducted at 75°C for 10 s. A 0.01wt% of sodium benzonate were added as a preservative. The juice was stored at -10°C and defrosted to room temperature before use and the distilled water were added to make up juice of the required concentrations. This experiment was carried out on a polysulfone hollow fiber membrane

module. They observed that the flux increased with temperature from 30 to 40°C and then decreased at 50°C. At low temperature the flux-pressure curves followed the gel-polarization model. The result were different at 50°C where flux initially increased with pressure and then subsequently decreased and that flux increased with increasing bulk flow rates and decreased with increasing bulk concentration, in accordance with concentration polarization models. Except for membrane resistance  $R_m$ , which was constant, other resistance increased with pressure and juice concentration and decreased with flow rate. An increase of temperature reduce the value of  $R_{p,re}$ , the reversible polarized layer resistance and  $R_{p,ir}$ , the semi-reversible polarized layer resistance but enhanced  $R_f$ , the fouling resistance.  $R_{p,re}$  was the major resistance which controlled the permeation flux for low temperature operation. At high temperature (50°C) the reversible polarized layer changed to a cross-linked gel and  $R_f$  was significantly increased.

The selection of the best condition for UF processing of passion fruit juice can be obtained from the results. If the maximum permeation flux is the requirement, the best condition tested would be at 40°C, 117 kPa of pressure and a flow rate of 1200 ml/min. The minimum fouling can be the requirement because the fouling gives rise to cleaning problems and shorten membrane life. For such case, the best conditions tested would be at 30°C, 68 kPa pressure and at a flow rate of 1200 ml/min. However, the results showed that at high flow rates the effect of pressure on  $R_f$  was not very significant and  $R_f$  at 30 & 40°C were slightly different. It would be appropriate to select the operating conditions for which the maximum flux is obtained.

Vaillant et al., 1999 studied Cross microfiltration after enzymatic liquefaction using ceramic membranes with 0.2  $\mu\text{m}$  pore size for clarification of passion fruit juice. Juice was taken from the industrial processing line after peel removal, enzymatic treatment of pulp (15 min) at

ambient temperature for the separation of the seeds from juice sacs, seed removal, juice finishing and centrifugation at 1000 g(1.8-2 min) in order to remove heavy starch granules. The effect of a high-rate enzymatic treatment for the degradation of suspended solids was assessed, resulting in the selection of a commercial enzymatic preparation. Partial enzymatic liquefaction of cell-wall polysaccharides prior to microfiltration provided an unusual pattern of flux increase after a short decline when cross flow velocity was high ( $7 \text{ m s}^{-1}$ ). It was found that a synergistic effect between pectinase and cellulase activities enhanced permeate flux with total recycling at  $36^{\circ}\text{C}$ , the combination of low transmembrane pressure (150 kPa) and high enzyme concentration ( $1 \text{ ml l}^{-1}$ ) provided the highest flux ( $113 \text{ l h}^{-1} \text{ m}^{-2}$ ). These conditions were then assessed with concentration in order to verify industrial feasibility and evaluate physicochemical characteristics of final products. A volumetric reduction ratio of 3 was maintained during 18 h without any decrease in permeate flux, which fluctuated around  $40 \text{ l h}^{-1} \text{ m}^{-2}$ . Retentate had similar characteristics of raw juice and could be recycled in order to use its residual enzyme activity.

Vaillant et al., 2005 they used osmotic evaporation instead of using reverse osmosis for the concentration of clarified juice. Melon juice is 1<sup>st</sup> clarified by cross flow microfiltration and then concentrated by osmotic evaporation (OE). The resulting clarified melon juice was highly similar to the initial juice, except for insoluble solids and carotenoids, which were concentrated in the retentate. Average permeation flux was relatively high (about  $80 \text{ L h}^{-1} \text{ m}^{-2}$ ), with continuous extraction of retentate at a volumetric reduction ratio of 3. After concentration of the clarified melon juice to as much as  $550 \text{ g kg}^{-1}$  of total soluble solids using a continuous feed-and-bleed procedure of OE, they found that almost the entire composition of the product was preserved. It preserves the main physico-chemical and nutritional properties. This integrated

membrane process permitted two valuable products to be obtained: a clarified concentrate of melon juice that had not undergone any thermal treatment, and a glowing-orange retentate that was enriched in provitamin A. Global yield of microbiological stabilized clarified juice of about 67%. The clarified juice presented physico-chemical and nutritional properties that were comparable with fresh melon juice, except for the absence of suspended solids and carotenoids, which remained totally concentrated in the retentate. The retentate presented a glowing orange colour because of the high concentration of h-carotene.

Reverse osmosis doesn't allow TSS as high as 300 of TSS  $\text{kg}^{-1}$  [Jariel et.al. 1996], OE appears to be an effective alternative. Also, it must be noted that the membrane used in these trials was not specific for OE. Trials carried out on a laboratory scale on sucrose solutions and with thinner membranes gave rise to much higher water evaporation fluxes of about  $10 \text{ l h}^{-1} \text{ m}^{-2}$  [Courel et.al.2000]. Also, it should be pointed out those porous organic hydrophobic membranes such as the one used here are cheap compared with the membranes used for reverse osmosis.

Cassano et al., 2003 evaluated the UF process for the clarification and concentration of carrot juice. Carrot juice was produced by chemical and physical treatment and it was supplied in frozen packages at pH 4.48 and with a concentration of 6 g TSS/100G. A limpid phase was produced in this step and it was used for concentration by successive membrane treatments. The RO process performed on a laboratory unit was used to preconcentrate the permeate coming from the UF step up to 15-20 g TSS/100g using Hydranautis spiral-wound membrane module composite polyamide membrane. A final OD step yielded a concentration of the retentate coming from RO up to 60-63g TSS/100g at an average throughput of about  $1 \text{ kg h m}^{-2}$ . This unit mainly

design to developed operational parameters for the design of a full-scale plant and secondly for the production of sample concentrate for their testing and evaluation.

The result obtained on the basis of these experimental tests concerning the identification of membrane module, the operating and fluid-dynamic conditions and cleaning procedures of membrane module. The performance of the membrane modules were evaluated on the basis of productivity, quality of the product and fouling characteristics. Advantages of the proposed integrated membrane system are in terms of: reduction of clarification times, simplification of the clarification process, increasing of clarified juice volumes, possibility to operate at room temperature preserving the juice's freshness, aroma and nutritional value, improvement of the quality of the final and improvement of the productive processes.

Gurak et al., 2010 evaluated the concentration of grape juice by reverse osmosis (RO). Preliminarily, a factorial design was carried out in which the independent variables were transmembrane pressure (40, 50 and 60 bar) and temperature (20, 30 and 40 °C) of the process, and the dependent variables were pH, content of soluble solids, acidity, concentration of phenolic compounds and those of monomeric and total anthocyanins, colour index, colour density, and permeate flux. None of the experiments resulted in significant changes in the juice characteristics. On the basis of high permeate flux value the best process conditions, 60 bar transmembrane pressure and 40 °C, was selected. They performed a new trial in order to determine whether increasing the temperature from 40 to 50 °C would result in any changes in the juice characteristics. The transmembrane pressure was kept at 60 bar, which was also the maximum value that could be applied by the equipment. Under these conditions, an increase in permeate flux was achieved with no significant difference in the physical or chemical parameters

of the product compared to the best condition corresponding to the factorial design. The physical and chemical properties of the concentrated juice increased in proportion to the volumetric concentration factor. It indicated the technical feasibility of reverse osmosis for pre-concentrating grape juice.

Grape juice can be concentrated up to 28.5 °Brix by reverse osmosis. The concentrated grape juice presented an increase in total titrable acidity, anthocyanin and phenolic compound contents, colour density and colour index proportional to the volumetric concentration factor. Reverse osmosis must be regarded as a pre-concentration process that can be coupled with other technologies to concentrate fruit juices up to 60° Brix as required by the fruit juice industry, while avoiding quality loss.

Mady et al., 2011 carried out an Osmotic Evaporation on grape juice. To prepare the juice were mixed with cooled demineralized water at a mass ratio of 1:5. After soaking for 3 h, the extract was filtered, first through a stainless steel sieve and then a polyester bag-filter system a micron rating of 5 µm. the extract were stored at 4°C. The industrial pilot plant used a hydrophobic, polypropylene, hollow-fiber membrane with an area of 10.2 m<sup>2</sup> and average pore diameter of 0.2µm. This was suitable for concentrating the fruit juice on various parameters such as temp, flow velocity and brine concentration. The final TSS contents achieved were 660g kg<sup>-1</sup> for grape juice. The physico-chemical, biochemical and aromatic qualities of concentrates obtained by OE were much higher than those thermal concentrates and close to those of the initial products.

Begona et al., 2009 studied the impact of high-pressure processing and temperature on onion juice nutritional attributes. Raw onions were hand peeled, washed with tap water at 4°C

for 1 min, rinsed with distilled water 30 s and dried. Quercetin and quercetin glucosides are the main onion flavonol. The experimental design comprised a response surface methodology according to a central composite face-centered design. The variable pressure were ranges 100-400 MPa and temperature 5-50°C, time was set up to 5 min.

They observed that the application of low temperature combined with pressures 100 and 400 MPa triggered to a better extraction of these flavonols among the treatment analyzed. Response surface of the antioxidant parameter as a function of pressure and temperature showed a clear trend towards an increase in onion antioxidant activity when applying pressure from 100 to 400 MPa. 400 MPa/5°C-processed onions showed an approximate 33% higher quercetin glucoside content compared with the untreated onion & maintain the antioxidant activity of the onion juice.

Drioli et al., 2011 investigated the production of concentrated pomegranate juice by using a two- step membrane process i.e. a clarification step of the fresh non-depectinized juice by hollow fiber poly (ether ether ketone) membrane and a concentration step of the clarified juice by OD of micro porous polypropylene membrane. For pretreatment fruit were washed in cold tap water and drained and they were manually cut up and the outer leathery skin which encloses hundreds of fleshy arils was removed and arils were pressed by using electric crusher, the extracted juice having a deep red color was prefiltered with a stainless steel filter, then it was stored in a refrigerator cell at -17°C. Both the process were performed at ambient temperature 25°C producing a clear juice and a concentrated juice with a total soluble solids content of 162 g kg<sup>-1</sup> and 520 g kg<sup>-1</sup> respectively. The performance of UD and OD evaluated in terms of productivity and quality of the processed juice. They observed that the suspended solids



completely removed in the clarification step, while soluble solids and organic acids were recovered in the permeate stream of the UF process. Rejection of the UF membrane towards polyphenols and anthocyanins of 16.5% and 11.7%. The antioxidant activity of pomegranate aril juice attributed to a great extent to total phenols and anthocyanins content was efficiently preserved during the concentration step independently on the achieved level of total soluble solids.

Matta et al., 2004 developed a process for obtaining clarified and concentrated acerola juice, maintaining its nutritional and sensory characteristics. A pulper machine with a 0.8 mm sieve was used for fruit pulping. The enzymatic hydrolysis of the pulp as a pretreatment for clarification were carried out in a mixture tank using the enzyme pectinex in the concentration of 0.01% (v/v), at 35°C, for 30min. This experiment consists of fruits pulping followed by an enzymatic hydrolysis, a clarification by MF and the concentration by RO. The product obtained of this experiment presented the required microbiological counts and nutritional quality due to the ascorbic acid preservation. MF reduced moulds, yeast and plate counting assuring a clarified juice adequate for consumption. Vitamin C content of the integral juice, 1234 mg/100 g, was maintained in the clarified juices. The concentrated juice had its vitamin C content improved in 4.2.folds reaching 5229 mg/100 g.

Timpone et al., 2007 studied the potentiality of a membrane-based process for the clarification and the concentration of the cactus pear juice. Fruits were manually washed in water and then peeled by hand, with a knife. Seed and mesocarp were removed with a squeezer and then washed with water and acid pectinase were added in quantity of 108g/kg and the puree was kept for 4 hrs at room temperature in plastic tanks without modification of the pH. It is first

clarified by UF of polysulfone membrane module with MWCO of 10 kDa and then concentrated by OD of polypropylene hollow fiber membrane with internal diameter 220  $\mu\text{m}$ . The clarification and concentration of the cactus pear juice by using UF and OD techniques represent a valid approach to process the juice at low temperature preserving the organoleptic, nutritional and sensorial characteristics of the fresh fruit. Analytical result of the UF process confirms the possibility to recover the most part of ascorbic, glutamic and citric acid in the clarified juice. The rejection of the UF membrane towards betaxanthins was lower than the rejection measured for betacyanins. Only 4% loss in the TAA was found in the UF permeate with respect to fresh juice. In the OD process the clarified juice with a TSS content of about 11°Brix was concentrated up to 61°Brix. An initial evaporation flux of 1.16 kg/m<sup>2</sup>h obtained using a calcium chloride dehydrates solution at 60 w/w% as stripper.

All the analytical parameters determined in the clarified juice remain constant during the process. In particular the final retentate of the OD process is a good source of antioxidant and it can be used in foods and nutritional formulations. Based on the above UF/OD integrated process permits to preserve the nutraceutical and functional importance of the cactus pear juice with respect to the traditional clarification and concentration procedures.

Cassano et al., 2007 modified poly(ether ether ketone) (PEEKWC) and polysulphone (PSU) hollow fiber (HF) membranes were prepared via dry-wet spinning technique through the phase inversion process and characterized by pure water permeability, dextran rejection and scanning electron microscopy (SEM). For clarification of Clementine mandarin juice the performance of the membranes, in terms of permeate flux and quality of permeate, was studied. Experiments were performed in selected operating conditions according to the batch

concentration procedure up to a final volume reduction factor (VRF) of 2. The permeate flux profile observed with the PSU membranes was lower than that observed with the PEEKWC membranes. Almost all total soluble solids and citric acid of the original juice were recovered in the permeate. After filtration the resulting juice showed a complete removal of suspended solids with a consequent improvement of colour and clarity. PEEKWC membranes showed a lower rejection towards antioxidant compounds (21.8%) in comparison with the PSU membranes (32.0%).

Saha et al., 2007 carried out clarification of sugarcane juice by UF and MF. Polysaccharides in sugarcane juice have an adverse impact on the manufacturing process and final sugar quality they also contribute to membrane fouling during ultrafiltration. Ultrafiltration (UF) of sugarcane juice with polysulphone (PS)/polyethersulphone (PES) membranes is characterized by significant fouling. Earlier they studied that a high molecular weight (HMW) component in the juice polysaccharide fraction was observed to interact with the membrane surface. This work examined fouling with sugarcane juice polysaccharides using polymeric PS/PES membranes in the 30–100 kDa range. For all these membranes, strong surface fouling is an important phenomenon. The HMW component present in the polysaccharide, containing arabinogalactan protein, along with some phenolic and lipids, appears to be primarily responsible for fouling. Surface fictionalization of PES UF membranes with thin grafted polymer hydro gel layers has recently been demonstrated to be a promising approach to obtain membranes with much higher fouling resistance to different classes of foulants such as proteins or polysaccharides.

Romero et al., 2009 worked on concentrating commercial noni juice (*Morinda citrifolia*) by Osmotic distillation (OD) or osmotic evaporation (OE) under isothermal conditions. Several nutraceutical properties have been reported for Noni derived products. In osmotic distillation system where the solutions are circulated through a hollow fiber membrane contactor operating in transient configuration with circulation rates between 0.1 and 1.0 L min<sup>-1</sup> and concentrated solutions of CaCl<sub>2</sub> were used as extraction brine. At isothermal conditions (30°C), transmembrane vapor water flux was experimentally determined from 0.090 up to 0.413 kg h<sup>-1</sup>m<sup>-2</sup>. After 60 min of treatment noni juice was concentrated from 8 to 32° Brix. The content of phenolic compounds was preserved after this processing. Simulation algorithms based on phenomenological equations of heat and mass transfer were developed considering a resistances-in-series model to predict the performance of the process from theoretical information. With the experimental ones for the operating conditions applied in this work the values of transmembrane water flux obtained by simulations showed deviations between 2.35 and 16.19%.

Petrotos et al., 2010 developed a novel membrane module for concentrating tomato juice by direct osmosis at ambient temperature and low pressure. This stainless steel module was in the shape of a flat configuration consisting of two parts of a square flange screwed together, with a piece of flat membrane between them. The configuration resulted in the formation of two chambers of special morphology which allowed the flow of tomato juice and osmotic medium in the two respective sides of the membrane and enabled the osmotic transfer of pure water from the juice to the osmotic medium side. Fresh tomato juice was concentrated from 5.5°Brix to the level of approximately 16°Brix. For re-concentration of the post-process diluted osmotic NaCl

brines, electrodialysis is proposed as a viable alternative to the commonly used evaporative process.

## CONCLUSION

Membrane technology gives information about extent of separation. With the investigation of membrane separation technology it was observed that the potential advantages of membrane concentration techniques over conventional evaporation for concentrating fruit juice have been successfully demonstrated, including improved product quality, easily scaled up and lower energy consumption, but they are generally limited by the fouling and lack of longer durability of membranes. Although today fruit juice concentration by membranes may be more expensive than evaporation. With the enlargement of the world's fruit juice market and the request of product quality, commercial application of membrane processes in concentrated fruit juice processing, especially integrated membrane system, will expand in the future. However, in order to gain a foothold in the juice industry, studies on developments of new membrane which are both highly selective and permeable or robust and stable in long-term application for juice processing and improvement of process engineering including module design and process design and optimization need to be carried out in detail. It can be anticipated that the use of membrane processes will bring great changes in the fruit juice industry in the future, with the development of membrane science and technology.

### *Abbreviations*

AIS	-	Alcohol Insoluble Solid
AFM	-	Atomic Force Microscopy
ANN	-	Artificial Neural Network

CJ	-	Centrifuged Juice
DCMD	-	Direct Contact Membrane Distillation
ETCJ	-	Enzyme Treated Centrifuged Juice
FAO	-	Food and Agricultural Organization of United States
HMF	-	Haze of Membrane Filtration
HF	-	Hollow Fiber
HMW	-	High Molecular Weight
HPLC	-	High Performance Liquid Chromatography
MWCO	-	Molecular Weight cut off
MF	-	Microfiltration
MD	-	Membrane Distillation
NTU	-	Number of Transfer Unit
OD	-	Osmotic Distillation
OE	-	Osmotic Evaporation
PV	-	Pervaporation
POD	-	Peroxidase
PVDF	-	Polyvinylidene Fluoride
VRF	-	Volume Reduction Factor
PEEKM	-	Poly Ether Ether Ketone Membrane
PESM	-	Polyethersulfone Membrane
RO	-	Reverse Osmosis
SEM	-	Scanning Electron Microscopy

TFC	-	Thin Film Composite
TSS	-	Total Soluble Solid
TMP	-	Trans Membrane Pressure
TAA	-	Total Antioxidant Activity
UF	-	Ultrafiltration
PSM	-	Polysulfone Membrane
$R_m$	-	Membrane Resistance
$R_{p,re}$	-	Reversible Polarized Layer Resistance
$R_{p,ir}$	-	Semi-Reversible Polarized Layer Resistance
$R_f$	-	Fouling Resistance

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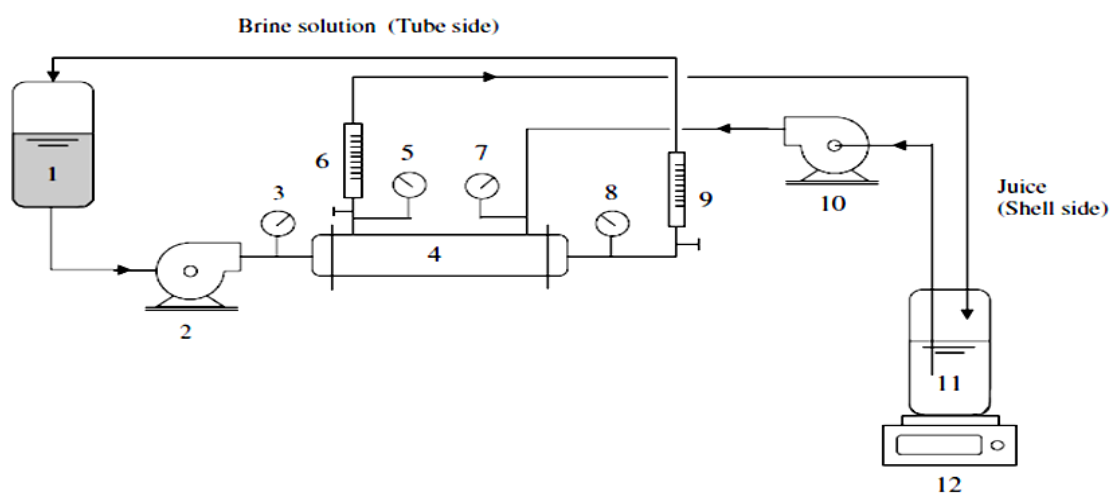


Fig.1: OD Experimental Set-up:-

Scheme of OD laboratory plant: 1. Extracting solution tank, 2. Brine pumps, 3,5,7,8. Manometers, 4. OD membrane module, 6, 9. Floemeters, 10. Feed pumps, 11. Feed tank, 12. Digital balance. (Onsekizoglu et al., 2010)

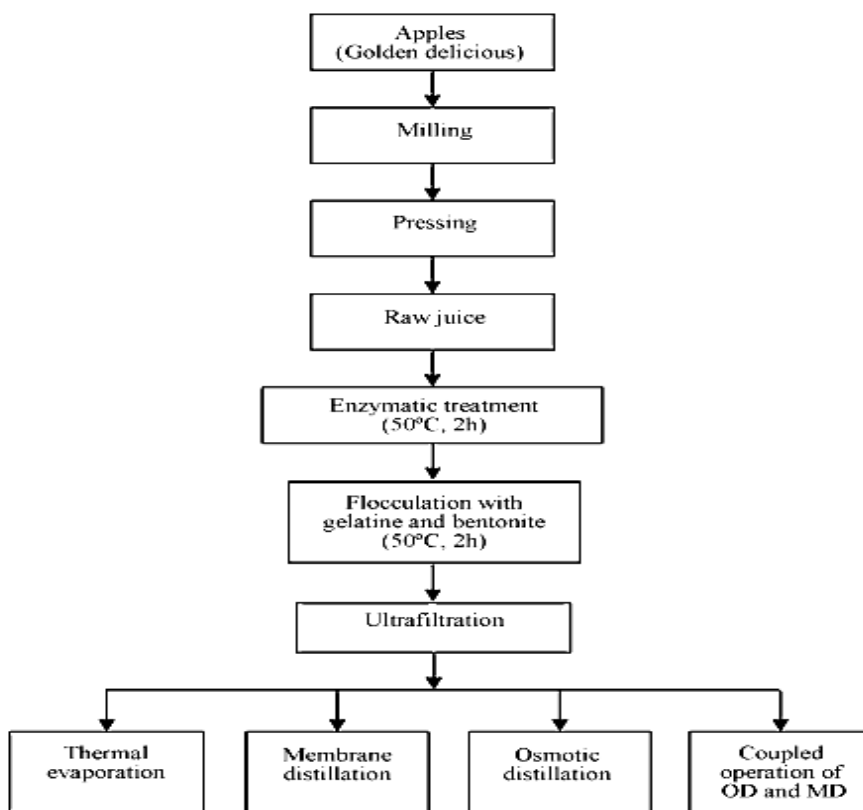


Fig.2. Laboratory scale clear apple juice concentrates processing scheme following different concentration techniques.

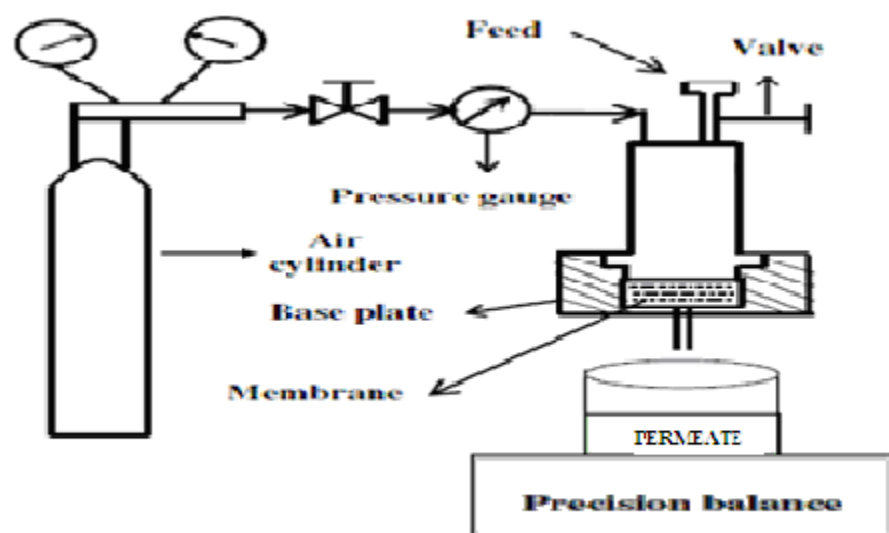


Fig.3. Schematic of experimental setup for the microfiltration experiment

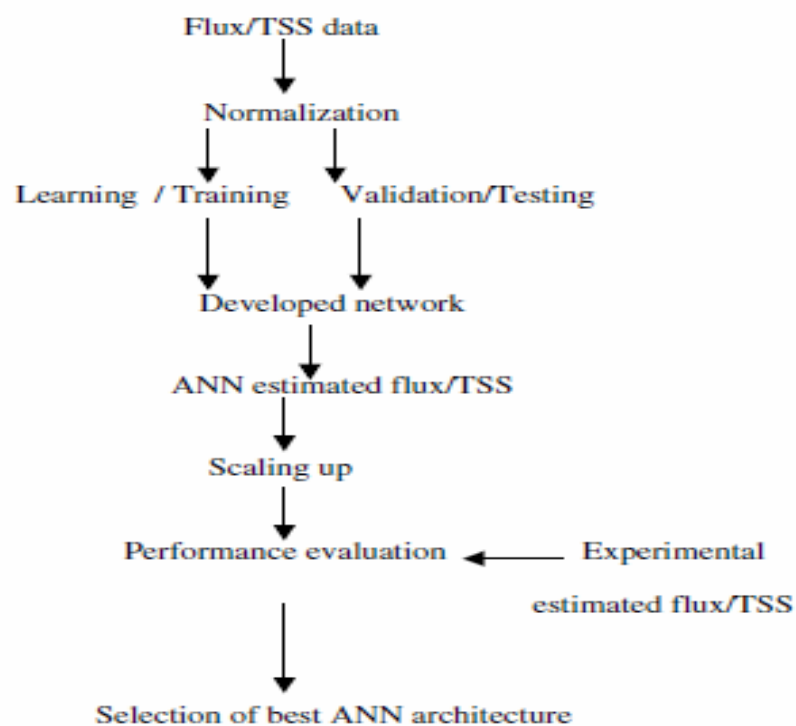


Fig.4 Methodology for developing ANN architecture.

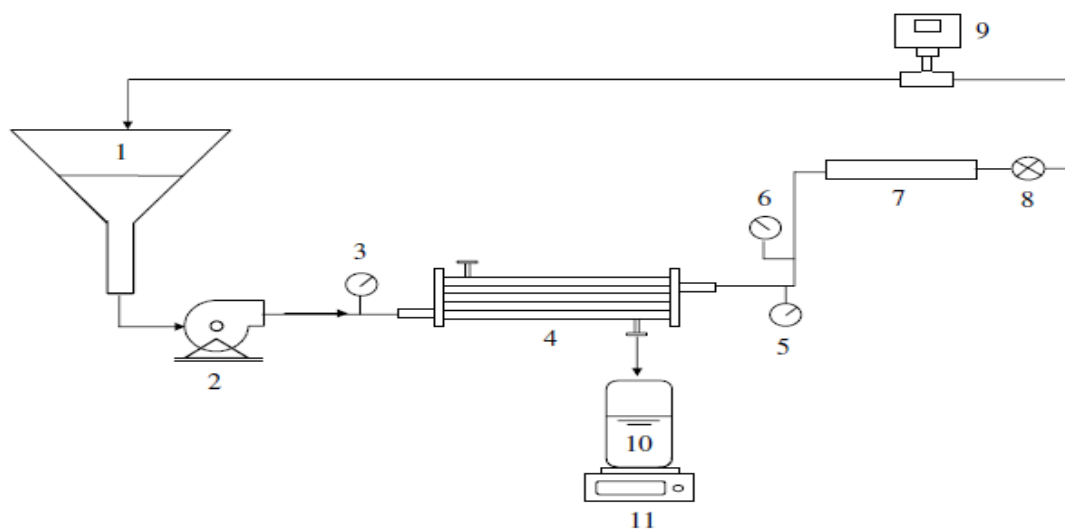
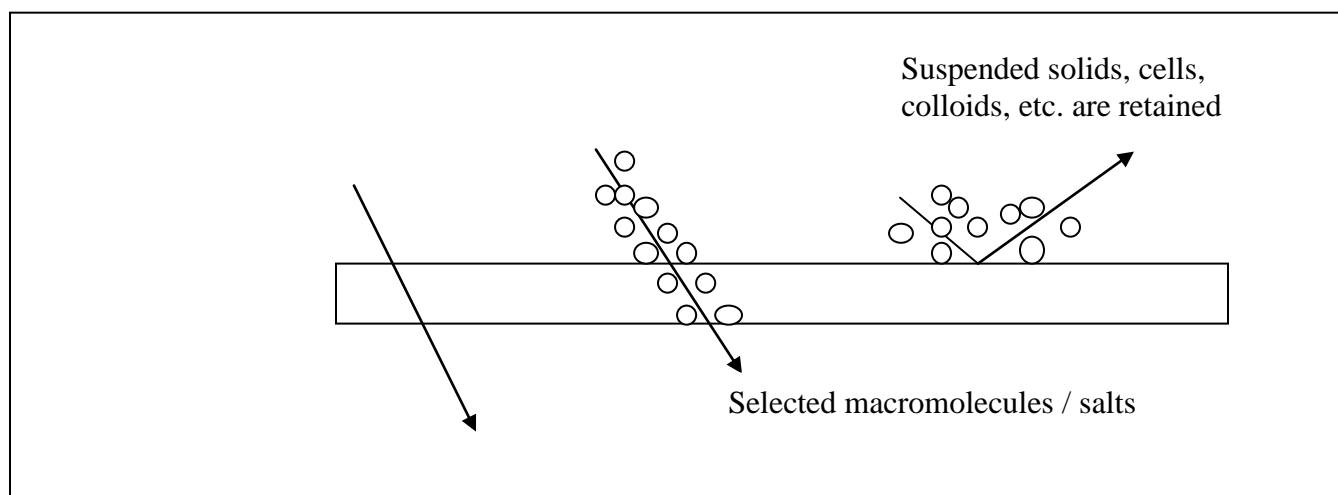


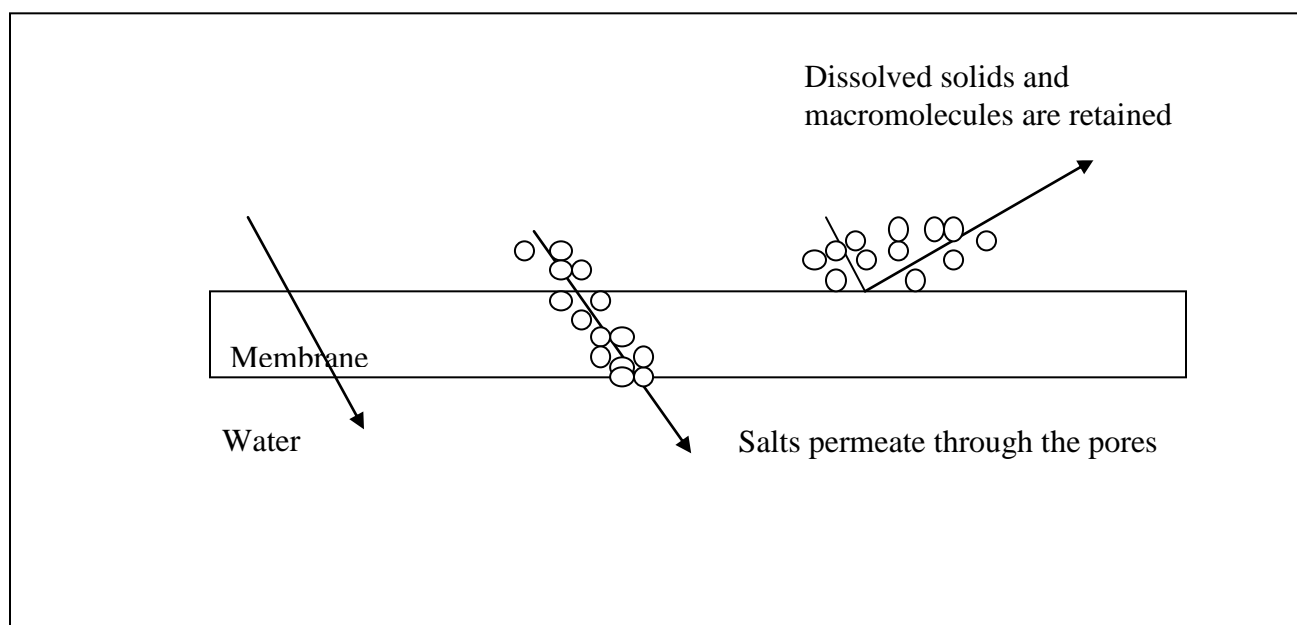
Fig.5. Scheme of the UF pilot laboratory plant (1 – feed tank; 2 – feed pump; 3, 6 –manometers; 4– membrane module; 5 – thermometer; 7 – heat exchanger; 8 – pressure valve; 9 – flow meter; 10 – permeate; 11 – digital balance).

**FIG. 6. SCHEMATIC REPRESENTATION OF COMMON MEMBRANE SEPARATION TECHNIQUES: (Dutta, 2007)**

**1) MICROFILTRATION**

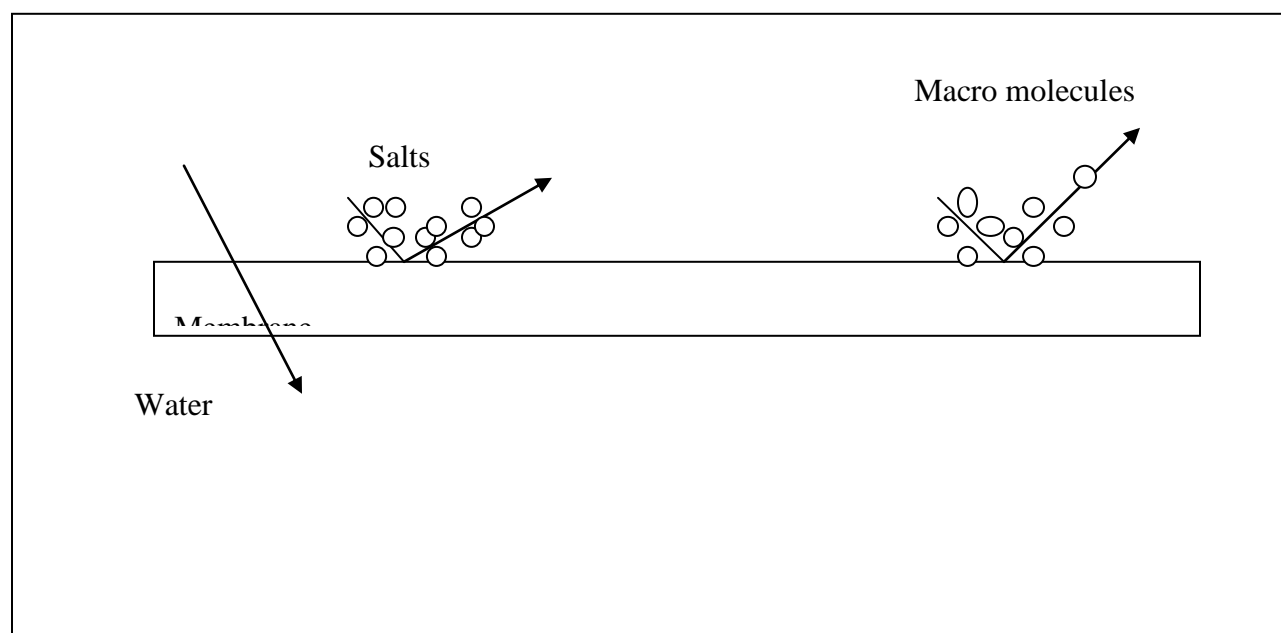


## 2) ULTRAFILTRATION





### 3) REVERSE OSMOSIS



**TABLE 1. SALIENT FEATURES OF VARIOUS MEMBRANE PROCESSES.**

(Cheryan, 1991; Ben and Gupta, 1990)

<b>Parameter</b>	<b>Ultrafiltration</b>	<b>Microfiltration</b>	<b>Reverse osmosis</b>
Membrane pore size, mm	1 - 50	100 - 2000	0.5 - 2
Operating pressure, atms	1-15	0.3-2.0	15-75
Permeate	Water + small molecules	Water + dissolved solutes	Water
Retentate	Most organic compound over 1000 mol.wt. including Viruses, Bacteria and Colloids	Large suspended particles, some emulsions, most bacteria	Ions and most organic compounds above 200 molecular wt.
Applications	Clarification and chillproofing of clear fruit juices. (Carvalho et al., 2008)	Clarification of apple juice. (Tajchakvit and Boye, 2001)	Concentration of fruit juice. (Cassano et al., 2003)

**TABLE 2. COMMON MEMBRANE REPORTED IN JUICE CONCENTRATION, CLARIFICATION AND FILTRATION.**

<b>Membrane Type</b>	<b>Application of fruit juice used</b>
Cellulose Acetate	Kiwifruit juice (MF), pineapple juice (UF).
Ceramic	Passion fruit, Mosambi fruit juice, (MF)
Polyamide	Apple juice(MF), Mosambi juice(UF), Carrot juice(RO)
Polycarbonate	Apple juice (UF)
Polyimide	Sugercane juice(UF)
Polyester	Mandarin juice(MF), Pomegranate juice(UF)
Polypropylene	Kiwifruit juice(MF), Noni juice( UF)
Polysulfone	Mosambi juice, Pineapple juice(MF), Orangejuice(UF),
PVDF	Carrot juice, Orange juice, Kiwifruit juice(UF),
PTFE	Mosambi juice, Apple juice(MF), Pineapple juice (UF).

**TABLE3. Literature on Fruit Juice Processing with Membrane**

<b>Sr. No</b>	<b>Application of Juice</b>	<b>Concentration Process</b>	<b>Parameter Studied</b>	<b>References</b>
1	Apple Juice	Vinylidene Fluoride Hydrophobic MF of DCMD of MFFK-3- Types	Concentration of apple juice	Gunko et al., 2006
2	Apple Juice	UF 10 kDa, Osmotic Distillation coupled with Membrane Distillation	Reduce tras-2-hexenal losses. Reduce haze formation	Onsekizogle et al., 2010
3	Apple Juice	NF of tubular membrane and two flat sheet membrane	feasible by use NF at low pressure & effect of morphology on the Conc. yield	Warczok et al., 2004
4	Apple juice	UF unit with tubular Carbosep M8 membrane consisting of a porous carbon support & active zirconium layer with MWCO 50 kDa	Blocking mechanisms of an UF by juice, using a recently developed membrane fouling model	Bruijn and Borquez, 2006
5	Clarified Apple Juice	UF through 100 kDa MWCO membrane at 50°C.	effect of modifying various processing condition, omission of amyloglucosidase, effect of diff.	Tajchakavit and Boye, 2001
6	Apple Juice	UF of Polysulphonamide Commercial membrane with MWCO 50,000Da	Fining treatments Change in natural polymer contents on juice Conc. By MD. & bentonite treatment followed by filtration	Lukanin et al., 2003
7	Clarified Apple Juice	UF of Carbosep membrane of 150 kDa	Membrane fouling, process efficiency and permeate flux	Alvarez and Riera, 1998

			varies between 56 & 157 L/(m <sup>2</sup> h)	
8	Mosambi Juice	UF with flat sheet Polythiersulfone membrane of MWCO 50 kDa	Effect of external D.C. electric field on the enhancement of permeates flux & estimation of gel polarization model	Sarkar et al., 2008
9	Mosambi Juice	UF thin film composite Polyamide of MWCO 10k,30k, 50k, & 100k	Effect of various cut off on permeate flux & quality of juice	Rai et al., 2007
10	Mosambi Juice	MF of Ceramic membrane	Properties such as colour, clarity, P <sup>H</sup> , citric acid, TSS and alcohol insoluble solid	Nandi et al., 2009
11	Mosambi Juice	UF with thin film composite polyamide membrane of MWCO 50,000 kDa	To predict the permeate flux & permeate concentration as total soluble solid during UF of fruit juice	Rai et al., 2005
12	Mosambi Juice	UF with flat sheet Polythiersulfone membrane of MWCO 50 kDa	Effect of enzyme treatment on juice & optimize the process condition by response surface methodology	Rai et al., 2005
13	Clarified Kiwifruit Juice	Polypropylene hollow fiber membrane with OD	Impact in terms of ascorbic acid content & (TAA). OD in terms of flux & concentration factor evaluated of the juice	Cassano et al., 2007
14	Kiwifruit Juice	Tubular PVDF with 15 kDa with OD, UF & PV	Effect of operation of TAA, ascorbic acid	Sindona et al., 2006

			content and aroma profile of the juice evaluated	
15	Kiwifruit Juice	Tubular polyvinyl-polypyrrolidine (PVPP) membrane	Effect of high pressure combined with heat treatment on kiwifruit peroxidase in food and model system	Fang et al., 2008
16	Kiwifruit Juice	Tubular membrane module of PVDF of 15 kDa	To study the effect of different operating parameters i.e. TMP, cross flow velocity & temp on permeate flux	Cassano et al., 2007
17	Kiwifruit Juice	Tubular membrane module of PVDF of 15 kDa with surface area 0.23m <sup>2</sup>	To design an integrated process for juice with high nutritional value	Cassano et al., 2004
18	Kiwifruit Juice	UF of cellulose acetate membrane of 30 kDa	Influence of bioactive composition like glutamic acid, citric, folic & ascorbic acid	Conidi et al., 2009
19	Orange Juice	Polysulphone/Polyethylene composite layer with RO	Quality of juice, the concentration factor, soluble solid content & vitamin C concentration	Jesus et al., 2007
20	Blood Orange Juice	Tubular PVDF membrane by UF of 15 kDa	Effect of TMP, axial feed-flow rate, temperature on permeate flux	Caasano et al., 2007
21	Orange Juice	Polysulphone membrane	Process of freeze concentration of juice using a multi-plate device	Sanchez et al., 2010
22	Blood Orange Juice	Tubular PVDF membrane by UF of 15 kDa. Surface membrane area 0.23m <sup>2</sup>	Behaviour of the different bioactive compounds & to	Gianni et al., 2008

			maintain TAA of the juice	
23	Pineapple Juice	MF of Polysulfone hollow fiber module with fiber dia.1 mm & length 30 cm	Effect of membrane pore size on permeate flux, fouling & quality of clarified juice	Laorko et al., 2010
24	Pineapple Juice	UF 50 kDa Polysulfone membrane with 0.3µm PES	At pressure 7.5 bar best sugar recovery 90.79%	Carvalho et al., 2008
25	Pineapple Juice	UF of cellulose Acetate with MWCO 30 kDa	To evaluate the inhibitory effect of chitoson/methyl cellulose film with vanillin against E. Coli	Sangsuwan et al., 2008
26	Passion Fruit	UF of hollow fiber module of polysulfone membrane of nominal MWCO of 30 kDa	Operating parameters on fouling during the UF. Analysis in terms of resistance to permeation flux	Jiraratananon and Chanachai, 1996
27	Passion Fruit	Cross flow MF(0.2µm) ceramic membrane, partial enzymatic liquefaction	Quality of permeate is satisfying, aromatic strength was weakened	Vaillant et al., 1999
28	Melon Juice	Clarified by cross flow MF then concentrated by OE feed and bleed procedure	Study the effect of CMF and OE on the physic-chemical, nutritional and microbiological qualities of melon juice	Vaillant et al., 2005
29	Carrot Juice	UF of tubular membrane module PVDF with MWCA 15 kDa and RO with composite polyamide, OD of micro porous polypropylene hallow fiber	To identify the integrated membrane process for carrot juice with high nutritional value	Cassano et al., 2003

Celgard membranes				
30	Grape Juice	RO with composite layer of polysulfone	To measure the concentration and characteristics of the grape juice	Gurak et al., 2010
31	Grape Juice	OE with a hydrophobic, polypropylene, hollow-fiber membrane with an area of 10.2m <sup>2</sup> & avg. Pore diameter of 0.2 μm.	Potential of using OE to concentrate the juice & impact on product quality	Maddy et al., 2011
32	Onion Juice	Response Surface Methodology	Effect of combined high pressure & temperature on phenol content, flavonol content & antioxidant activity	Begona et al., 2009
33	Pomegranate Juice	UF of modified poly (ether ether ketone) hollow fiber module with dia. 1.64 mm & OD of micro porous polypropylene HF with inner dia. 300μm	Recovery of TSS content of 162 g.kg <sup>-1</sup> & 520 g.kg <sup>-1</sup>	Drioli et al., 2011
34	Acerola Juice	MF unit of three tubular membranes of polyethersulfone pore size of 0.3μm with a permeation area of 0.05m <sup>2</sup>	To evaluate the sensory, microbiological and nutritional properties of the juice	Matta et al., 2004
35	Cactus Pear Juice	UF of Ploysulfone membrane module with MWCO 10 kDa & pressure 2.1 bar at 25°C	Analytical measurement of the parameters which characterize the high nutritional and organoleptic properties of juice	Timpone et al., 2007
36	Clementine Mandarin Juice	UF poly ether ether ketone and polysulfone hollow fiber membrane	To improve the colour & clarity of the juice	Cassano et al., 2007
37	Sugarcane Juice	UF of hollow fiber module of polysulfone membrane	Impact on manufacturing	Saha et al., 2007



		of nominal MWCO of 30 kDa	process & membrane fouling during UF	
38	Noni Juice	OE with a hydrophobic, polypropylene, hollow-fiber membrane	Potential of using OE to concentrate the juice & impact on product quality	Romero et al., 2009
39	Tomato Juice	Polypropylene hollow fiber membrane with Direct Osmosis	To measure the concentration of the tomato juice	Petrotos et al., 2010
40	Orange Juice	RO membrane of Aromatic polyamide	Fouling mechanism and flux decline mechanism	Mayor et al., 2011

**TABLE 4. Advantages and Disadvantages of RO applied to the concentration of fruit juice. (Jaio et al., 2004)**

<ol style="list-style-type: none"> <li>1. Broad industrial scale application</li> <li>2. Low temperatures</li> <li>3. Combination with vacuum evaporation and with system of vapour recompression</li> <li>4. Commercially available</li> <li>5. Energetically and economically convenient in comparison with the thermal evaporation</li> </ol>	<ol style="list-style-type: none"> <li>1. Fouling phenomena</li> <li>2. High pressures</li> <li>3. Necessity of an inactivation enzyme pre-treatment</li> <li>4. Juice concentration limited at 22-23°Brix</li> <li>5. Loss of aroma compounds during the process</li> <li>6. Membrane replacement at high cost</li> <li>7. Difficulty to concentrate solutions with high level of suspended solids</li> </ol>
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**TABLE 5. Advantages and Disadvantages of MD and OD applied to the concentration of fruit juice. (Jaio et al., 2004)**

Advantages	Disadvantages
<ol style="list-style-type: none"> <li>1. Techniques suitable for heat sensitive products</li> <li>2. A 60% dry matter can be reached in the concentrated product</li> <li>3. Modularity, easy scale-up</li> <li>4. Possibility to treat solutions with high level of suspended solid</li> <li>5. Low temperature</li> <li>6. Low operating pressure</li> <li>7. No fouling problem, constant permeate flux in time</li> <li>8. New technologies based on the use of conventional well-tested materials</li> <li>9. Low investment cost</li> </ol>	<ol style="list-style-type: none"> <li>1. New technology that requires an evaluation at industrial level, flexibility to be evaluated</li> <li>2. Low evaporative capacity (3 l/m<sup>2</sup>h) with a long time of treatment</li> <li>3. Necessity of an inactivation enzyme pre-treatment</li> <li>4. Production cost higher than the thermal evaporation</li> <li>5. High cost of membrane replacement</li> </ol>

**TABLE 6. ENERGY REQUIRMENT IN VARIOUS FILTRATION PROESSES.****(Riley et al., 1967; Timpone et al., 2007)**

<b>Filtration Process</b>	<b>Device Type</b>	<b>Energy Savings</b>
Reverse Osmosis	Energy Recovery Turbine	30 – 40 %
	Pressure Exchanger	50 – 60 %
Ultrafiltration	Energy Recovery Turbine	40 – 50%
	Pressure Exchanger	60 – 68 %
Microfiltration	Energy Recovery Turbine	65 – 75 %
	Pressure Exchanger	75 – 80 %
Diafiltration	Energy Recovery Turbine	45 -65 %
	Pressure Exchanger	65 – 70%