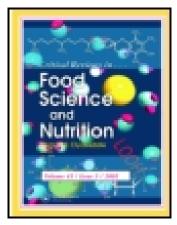
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# Opportunities and challenges in application of forward osmosis in food processing

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#### Opportunities and challenges in application of forward osmosis in food processing

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#### **Abstract**

Food processing and preservation technologies must maintain the fresh like characteristics of the food while providing an acceptable and convenient shelf life as well as assuring safety and nutritional value. Besides, the consumers demand for highest quality convenience foods in terms of natural flavor and taste, free from additives and preservatives necessitated the development of a number of membrane based nonthermal approaches to the concentration of liquid foods, of which forward osmosis has proven to be most valuable. A series of recent publications in scientific journals have demonstrated novel and diverse uses of this technology for food processing, desalination, pharmaceuticals as well as for power generation. Its novel features, which include the concentration of liquid foods as at ambient temperature and pressure without significant fouling of membrane made the technology commercially attractive. This review aims to identify the opportunities and challenges associated with this technology. At the same time, it presents a comprehensive account of recent advances in forward osmosis technology as related to the major issues of concern in its rapidly growing applications in food processing such as concentration of fruits and vegetables juices (grape, pineapple, red raspberry, orange, tomato Juice and red radish juices) and natural food colorants (anthocyanin and betalains extracts). Several vibrant and vital issues such as recent developments in the forward osmosis membrane and concentration polarization aspects have also been addressed. The asymmetric membrane used for forward osmosis poses newer challenges to account both external and internal concentration polarization leading to significant reduction in flux. The recent advances and developments in forward osmosis membrane processes, mechanism of water transport,

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characteristics of draw solution and membranes as well as applications of forward osmosis in food processing have been discussed.

Key words: Forward/direct osmosis, membrane, hydrophilic, osmosis, desalination

#### 1. Introduction

Osmosis is a physical phenomenon that has been extensively studied by scientists and researchers in various disciplines of science, engineering and technology. Osmosis phenomenon is defined as a net movement of water across a selectively permeable membrane driven by a difference in osmotic pressure across the membrane. Liquid foods such as fruit juices are one of the important sources of bioactive compounds including vitamins, phenolic compounds and pigments such as anthocyanins and carotenoids. Fruit juices are generally concentrated to achieve longer shelf-life by employing thermal evaporation under vacuum in order to reduce the storage and transportation costs. It leads to the loss of fresh juice flavors and color degradation besides development of cooked taste due to the thermal treatment. The use of thermal processing technologies such as vacuum evaporation and subsequent storage conditions may cause significant changes in the bioactive compounds. Similarly, the natural color extracts when extracted from the plant source are of very dilute nature due to presence of water as a major constituent and the colorant concentration is of the order of 0.1% or less (Thomas, 1984). It needs to be concentrated to avoid growth of the microorganisms, improve shelf-life and stability as well as to reduce storage and transportation costs. The concentration of natural color extracts by conventional method namely evaporation results in loss of hue and chroma resulting in low quality product (Bhaskaran and Mehta, 2006; Patil and Raghavarao, 2007).

Increasing consumer demand for good quality fruit juices with natural flavor and taste, free from additives and preservatives as well as realizing the need for the non-thermal processing techniques for concentration of natural colorant stimulated the need for the development of innovative approaches for concentration in order to produce concentrated products with a minimum loss of nutritional, physicochemical, or organoleptic properties (Babu *et al.*, 2006).

Evaporative concentration of fluid food results in heat induced deterioration of sensory and nutritional quality of finished product. The first few minutes of evaporative concentration results in loss of most of the aroma compounds present in raw juice (Lazarides *et al.*, 1990). Use of vacuum evaporation for juice concentration is highly energy intensive (Petrotos and Lazarides, 2001). Product quality improvement and energy savings had lead to the development of alternative concentration techniques such as freeze concentration or reverse osmosis. The capital and operating costs of a commercial scale freeze concentration plant are much higher than the cost of an evaporation plant for a similar throughput. Membrane processes such as microfiltration, ultrafiltration and reverse osmosis were being employed for clarification and concentration of fruit juice as well as natural color extracts. There are many limitations of these membrane processes such as requirement of high pressure, limit to maximum attainable concentration, concentration polarization and membrane fouling (Rastogi and Nayak, 2011).

Forward osmosis is regarded as an innovation in the field of membrane separation, which allows the concentrating juices with higher concentration of both dissolved and suspended solids, without significant fouling of the membrane (Beaudry and Lampi, 1990a). It is an osmotically driven membrane separation process that relies on the utilization of a large osmotic pressure differential generated across a semi-permeable dense hydrophilic membrane that separates two

aqueous solutions (feed and osmotic agent solution) having different osmotic pressures. The difference in osmotic pressure acts as a driving force. Osmotic pressure driven process operates on the principle of osmotic transport of water across a semi-permeable hydrophilic membrane from a dilute feed solution into a concentrated draw solution (Nayak and Rastogi, 2010a). The forward osmosis process is also known as 'direct osmosis' or in the case of a number of companies who have coined their own terminology such as 'engineered osmosis' and 'manipulated osmosis'. It is a membrane process capable of concentrating liquid foods at ambient or cooler temperatures. The process utilizes a draw solution to separate water from liquid foods through a semi-permeable membrane. The draw solute must not pass through the membrane and should have an osmotic pressure greater than the osmotic pressure of the concentrated fruit juice. In contrast, the reverse osmosis process uses hydraulic pressure as the driving force for separation, which serves to counteract the osmotic pressure gradient that would otherwise favor water flux from the permeate to the feed.

With the explosion of global population, enhanced water pollution and standard of living have inexorably put pressure on water and energy resources. Low cost methods of purifying freshwater and desalting seawater are required to compete with this destabilizing trend. Production of purified water from saline water using membranes has become a viable alternative to evaporation-based technologies. The concept is similar to that used by Eastern European farmers for the concentration of fruit juices, in which juice filled bag was subjected to brine solution leading to a highly concentrated juice. But it took a long time to concentrate juices (Cussler, 1984). A commercial process involving liquid food (fruit juices, milk or dairy products) filled in a bag made of semi-permeable material (cellulose, polysulphones or animal intestines)

was subjected to a bath of osmotic medium at an elevated temperature. Further, higher water flux and a significant reduction in time were obtained by continuously stirring the bag (Scott, 1975).

The pioneering work for the concentration of fruit juice by forward osmosis process was reported by Popper *et al.* (1966) using first generation reverse osmosis membranes (cellulose acetate) in both flat and tubular configurations. However, the average transmembrane flux was lower (~2.5 L.m<sup>-2</sup>.h<sup>-1</sup>). New generation modified thin film composite reverse osmosis membranes (thickness 25–85 μm) used by Beaudry and Lampi (1990a, b) indicated that no passage to other food ingredients except water was allowed. Further, the modified membrane resulted in higher turbulence, osmotic flux (5-6 L.m<sup>-2</sup>.h<sup>-1</sup>) and reduced fouling (Herron *et al.*, 1994). Subsequently, Petrotos *et al.* (1998, 1999) indicated that a thinner membrane and a low viscosity osmotic medium (such as NaCl solution) showed a better performance as compared to viscous draw solution. The clarification of the juice either by filtration (microfiltration or ultrafiltration) resulted in higher fluxes. Later, an osmotic cell of flat configuration was developed to promote turbulence leading to higher average flux (4.5 L.m<sup>-2</sup>.h<sup>-1</sup>) by Petrotos and Lazarides (2001). The forward osmosis concentrate was demonstrated to be of superior quality in comparison to juice conventionally concentrated by vacuum evaporator (Herron *et al.*, 1994).

In recent years, forward osmosis concentration is gaining importance for the concentration of liquid foods and natural colours (Popper *et al.*, 1966; Bolin and Salunke, 1971; Loeb and Bloch, 1973; Rodriguez-Saona *et al.*, 2001; Babu *et al.*, 2006; Nayak and Rastogi, 2010a, b; Nayak *et al.*, 2011; Zhao *et al.*, 2012), waste water treatment (Holloway *et al.*, 2007), desalination of seawater as well as electricity generation (Kravath and Davis, 1975).

The main advantage of using forward osmosis is that it can concentrate feed at ambient pressure and temperature without significantly losing nutritional and bioactive components. Other advantages include reduced energy consumption, higher product quality in terms of a less loss of fresh fruit flavors, reduced color degradation and less cooked taste due to less thermal effects. There is no hydraulic pressure difference applied across the membrane and the dissolved as well as suspended solids in the feed are not forced against the membrane, so the membrane does not foul to a significant extent. It also results in high rejection of a wide range of contaminants and lower membrane fouling propensity than pressure-driven membrane processes (Babu et al., 2006). The pressure involved in the forward osmosis process is due to flow resistance in the membrane module, which involves the use of simple equipment (Cath et al., 2005a, b). In comparison to pressure-driven membrane processes such as reverse osmosis, forward osmosis uses significantly less electrical energy, if the osmotic agent solution can be easily recovered or discarded using less or lower quality energy (Garcia-Castello et al., 2009). The recovery or disposal osmotic agent solution may incur high energy and/or capital costs, which are considered as a necessary evil, which require a proper address before this technology can be used on a wider scale (McCutcheon et al., 2005).

Wong and Winger (1999) reviewed the applications of forward osmosis for food processing. Fruit juices can be concentrated to a higher concentration than with reverse osmosis, even with high insoluble solid levels and fouling has been reported to be minimal. The fluxes obtained with forward osmosis at present limit its full commercial acceptance in comparison to reverse osmosis. Most of the information available for the concentration of beverages and liquid foods using forward osmosis are limited to laboratory scale (Raghavarao *et al.*, 2005).

The capability and limitations of forward osmosis have been extensively reviewed (Jiao et al., 2004; Cath et al., 2006; McCutcheon and Elimelech, 2007; Wilf 2010; Rastogi and Nayak, 2011; Chung et al., 2012). Many of the early reviews primarily focused the perspectives on future R&D for forward osmosis processes in order to develop effective and sustainable technologies for water, energy and pharmaceutical production. This review comprehensively exclusively covers the various perspectives and its advantages of forward osmosis with special reference to food processing. The recent advances and developments in forward osmosis membrane processes, mechanism of water transport, draw solution characteristics, membranes for forward osmosis as well as applications of forward osmosis in food processing have been discussed in detail.

#### 2. Mechanism of forward osmosis

Osmosis is the movement of solvent molecules through a selectively permeable membrane into a region of higher solute concentration, aiming to equalize the solute concentrations on both the sides. Net movement of solvent is from the less concentrated (hypotonic) to the more concentrated (hypertonic) solution, which tends to reduce the difference in concentrations. When a semi-permeable membrane separates two solutions, water always diffuses from the solution with the lower osmotic potential to the solution with the higher osmotic potential. This diffusion-driven motion of water through a membrane is termed as 'osmosis'. It is the tendency of fluids to pass through a membrane so that equal concentrations are achieved on both the sides (Figure 1a). Reverse osmosis is pressure driven membrane process which entails forcing fluids through a membrane. It is a separation technique, which can be used to concentrate or purify liquids without a phase change. The reverse osmosis process uses

hydraulic pressure as the driving force for separation, which serves to counteract the osmotic pressure gradient that would otherwise favor water flux from the permeate to the feed (Figure 1b). Forward osmosis is an osmotic process that, like reverse osmosis, uses a semi-permeable membrane to effect separation of water from dissolved solutes. The driving force for this separation is an osmotic pressure gradient, such that a draw solution of high concentration (relative to that of the feed solution), is used to induce a net flow of water through the membrane into the draw solution, thus effectively separating the feed water from its solutes. Forward osmosis uses the osmotic pressure differential ( $\Delta\pi$ ) across the membrane as the driving force for transport of water through the membrane, rather than hydraulic pressure differential (similar to the reverse osmosis). The forward osmosis process results in concentration of feed solution and dilution of draw solution (Figure 1c). Pressure retarded osmosis can be considered as a process between forward osmosis and reverse osmosis, where hydraulic pressure is applied in the opposite direction of the osmotic pressure gradient (similar to reverse osmosis). However, the net water flux is still in the direction of the concentrated draw solution (similar to forward osmosis, Figure 1d) (Cath et al., 2006; Rastogi and Nayak, 2011). The simplest equation describing the relationship between osmotic and hydraulic pressures and water flux is:

$$J_{w} = A(\Delta \pi - \Delta P) \qquad \dots (1)$$

Where,  $J_w$  is the water flux, A is the water permeability of the membrane, and  $\Delta P$  is the difference in pressure (Xu *et al.*, 2010).

Concentration polarization is a significant problem in pressure-driven membrane processes such as reverse osmosis. It reduces permeate flow due to build up of the retained molecules leading to enhanced osmotic pressure at the membrane surface. The forward osmosis

phenomenon with a dense symmetric membrane can result in the occurrence of concentration polarization on both sides of the membrane. The solute is concentrated and diluted on the feed and permeate side, leading to concentrative and dilutive external concentration polarization, respectively (Figure 2a). The standard flux equation for forward equation is given by the following equation:

$$J_{w} = A(\pi_{d} - \pi_{f}) \qquad \dots (2)$$

Equation (2) may be valid only if the permeate flux is very low. For higher flux rates, the equation must be modified to the following equation to account for the dilutive and concentrative (feed/draw side) external concentration polarizations (McCutchen and Elimelech, 2007).

$$J_{w} = A(\pi^{*}_{d} - \pi^{*}_{f}) = A\left[\pi_{d} \exp\left(-\frac{J_{w}}{k_{d}}\right) - \pi_{f} \exp\left(\frac{J_{w}}{k_{f}}\right)\right] \qquad ....(3)$$

Where  $\pi_d$  and  $\pi_f$  are the bulk osmotic pressures of draw and feed solutions, respectively;

$$\pi^*_{_d} = \pi_{_d} \exp \left( -\frac{J_{_w}}{k_{_d}} \right) \text{ and } \pi^*_{_f} = \pi_{_f} \exp \left( \frac{J_{_w}}{k_{_f}} \right) \text{ the osmotic pressures on membrane surface of } m_{_f} = m_{_f} \exp \left( \frac{J_{_w}}{k_{_f}} \right)$$

draw and feed solutions, respectively;  $J_w$  is the water flux;  $k_d$  and  $k_f$  are the mass transfer coefficients on draw and feed solution sides, respectively.

The asymmetric membrane used in forward osmosis consisted of two layers, one is the loosely bound support layer and other is the dense active membrane layer. The membrane can be placed between the feed and the osmotic agent solutions in two different ways such that feed towards the support layer (normal mode) and feed towards active layer (reverse mode), which are referred as mode I (Figure 2b) and mode II (Figure 2c), respectively (Gray *et al.*, 2006; Nayak and Rastogi, 2010a).

When the feed is placed against the support layer (mode I), water enters the porous support layer and diffuses across the active layer into the draw solution. The salt present in feed along with water enters the open structure and transported into this layer by convective water flow. Since, the salt cannot pass easily pass through the active layer, it results in an increase in concentration within the support layer. This phenomenon is referred to as concentrative internal concentration polarization. It is assumed that in case of mode I no concentrative external concentration polarization occurs on the support layer because the support layer creates no hydraulic resistance to water transport and solute may freely enter the support layer. It has been pointed out by Cath *et al.* (2006) that in this situation the extent of external polarization is much less than the internal polarization during forward osmosis. On the permeate side of the membrane, dilutive external concentration polarization takes place due to displacement of dissolved draw solute away from the membrane surface, which lessens the effective driving force of the draw solution (Figure 2b).

If the feed solution is kept towards the active layer (mode II), the internal concentration polarization phenomenon occurs on the permeate side resulting in dilution of permeate water within the porous support of the membrane, which is termed as dilutive internal concentration polarization. The permeation of water from feed to draw solution side results in higher concentrations of solute near the active layer of membrane, which increases the osmotic pressure leading to the reduction in the driving force. It is termed as concentrative external concentration polarization. No external concentration polarization occurs at the draw side in case of mode II due to the same reason cited for the no external concentration polarization occurring in case of mode I on the feed side (Figure 2c). The internal concentration polarization occurs within the

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porous support layer and it cannot be mitigated by hydrodynamics such as turbulence, and hence drastically reduces the osmotic driving force (McCutcheon and Elimelech, 2007).

The equations (4) and (5) were found to model flux for forward osmosis through an asymmetric membrane in a situation when the feed is towards the support layer (mode I) and when feed is towards the active layer (mode II), respectively.

$$J_{w} = A[\pi *_{d} - \pi'_{f}] = A[\pi_{d} \exp(-\frac{J_{w}}{k_{d}}) - \pi_{f} \exp(J_{w}K_{f})]$$
 ....(4)

$$J_{w} = A[\pi'_{d} - \pi^{*}_{f}] = A \left[\pi_{d} \exp(-J_{w}K_{d}) - \pi_{f} \exp\left(\frac{J_{w}}{k_{f}}\right)\right] \qquad ....(5)$$

 $\pi'_f$  and  $\pi'_d$  are the osmotic pressures of the feed and draw solutions on the inside of the active layer within the porous support for concentrative internal concentration polarization on feed side and dilutive internal concentration polarization on draw side for mode I and mode II, respectively;  $\pi'_d = \pi_d \exp(-J_w K_d)$  and  $\pi'_f = \pi_f \exp(J_w K_f)$ ;  $K_f$  and  $K_d$  are the solute resistivity for diffusion within the porous support layer for mode I and mode II, respectively. It may be noted that in case of mode I, concentrative internal concentration polarization and dilutive external concentration polarization occur in pair. Similarly, for mode II, dilutive internal concentration polarization and concentrative external concentration appear together.

Nayak and Rastogi (2010a) described the mechanism of water transport in forward osmosis in a situation when feed solution was a mixture of low and high molecular weight compounds. High molecular weight compounds in mode I led to the buildup of the retained concentration of high molecular weight compounds on the support layer resulting in significant external concentration polarization on the feed side. Further, low molecular weight compounds

resulted in significant concentrative internal concentration polarization within the support layer. But, the external polarization towards draw side was negligible. In case of mode II, when feed (solution containing high and low molecular weight compounds) was kept towards active layer, the water from the feed was diffused into the active layer, which, in turn, was diffused to the support layer and then to the bulk through the boundary layer. The external polarization on feed side was taken as negligible in comparison to internal polarization as well as the molecular weight of the compounds in feed did not have any effect on transmembrane flux. When the solution of low molecular weight compounds was taken as a feed, the mode I was most desirable. However, when the solution of high molecular weight compounds is taken as a feed, mode II gave higher driving force and which, in turn, led to higher transmembrane flux (Nayak *et al.*, 2011).

Mi and Elimelech (2008) also indicated that these high molecular weight compounds may be deposited within the porous structure of the membrane leading to cake layer formation due to lack of shear force as well as hindered back diffusion in the porous structure. Further, Mi and Elimelech (2010) studied the fouling and cleaning behavior of forward osmosis membrane. The fouling of forward osmosis membrane due to alginate was reported to be almost fully reversible, with more than 98% recovery of permeate water flux possible after a simple water rinse without any chemical cleaning reagents. The fouling reversibility of forward osmosis membrane was attributed to the less compact organic fouling layer formed due to the lack of hydraulic pressure.

The resistivity of the membrane (support and active layer) during forward osmosis process for mode I and II could be calculated as per the following equation presented for

concentrative or dilutive internal concentration polarization, respectively (Gray *et al.*, 2006; McCutcheon and Elimelech, 2007; Tan and Ng, 2008; Loeb *et al.*, 1997).

$$K_{c} = \left(\frac{1}{J_{w}}\right) \ln \left(\frac{B + A\pi_{d} - J_{w}}{B + A\pi_{f}}\right) \qquad \dots (6)$$

$$K_{d} = \left(\frac{1}{J_{w}}\right) \ln \left(\frac{B + A\pi_{d}}{B + J_{w} + A\pi_{f}}\right) \qquad \dots (7)$$

where  $J_w$  is the transmembrane flux during forward osmosis,  $K_c$  and  $K_d$  are the resistivity of membrane (support and active layer) (s/m) for mode I and II, respectively;  $\pi_d$  and  $\pi_f$  are the osmotic pressure of draw and feed solutions, respectively. The constant 'A' (0.027 m/atm-day) and 'B' (0.011 m/day) refer to the water and solute permeability coefficients of the active layer of the membrane, respectively (Gray *et al.*, 2006; Leob *et al.*, 1997).

Tang et al. (2010) demonstrated that the forward osmosis flux was highly non-linear with respect to the apparent driving force (the concentration difference between the draw solution and the feed) due to internal concentration polarization. When active layer was facing draw solution (mode I) resulted in more severe internal concentration polarization as compared to the situation when the active layer faced the feed water (mode II). The loss of flux was reported to be due to the combined effects of the internal clogging of the forward osmosis support structure and enhanced internal concentration polarization in the support layer. Li et al. (2011a) proposed a novel approach to study the effect of the porous support structure on the internal concentration polarization with more degrees of freedom. The support layer was approximated by a well-defined network, which provided a spatial domain of the topological structure for numerically evaluating the convective diffusion in the porous media associated with the finite element analysis. Sagiv and Semiat (2011) also developed a steady-state finite element model, which was

an efficient quantitative tool for optimizing a given forward osmosis system. The model involved mass and momentum transport equations, which were solved for boundary conditions. It was found that a major reduction in water flux resulted mainly from resistance due to the draw side concentration polarization layer that in some cases may be higher than the concentration drop through the membrane skin which was proportional to the driving force. Gruber *et al.* (2011) simulated forward osmosis systems by computational fluid dynamics using the models for pressure-driven systems and the general analytical theory for flux modeling in asymmetric membranes. Zhao and Zou (2011a, b) demonstrated that internal concentration polarization in the support layer was strongly dependent on the physicochemical properties of the solution facing the support layer. Zhao *et al.* (2011) investigated the effect of membrane orientations on forward osmosis performance. In a situation when the feed was facing active layer, it resulted in more stable and higher water flux than that of the situation when support layer was towards the feed.

#### 3. Draw solutions for forward osmosis

One of main components for successful development of forward osmosis technologies is the selection of draw solution. The draw solution should fulfill the several criteria to be an optimal solution. It should have a higher osmotic pressure than the feed solution to produce high water flux and the reverse diffusion of the draw solution should be minimal. Reverse salt diffusion results in diffusion of trace quantities of salts through the membrane from the draw solution into the feed solution, which reduces the driving force and may contaminate the feed solution and degrade the quality of the concentrated product. Another important criterion is the availability of a suitable process for effective and economical reconcentration of the draw

solution. For instance, when considering forward osmosis for production of potable water, it is important that draw solutes are not present in the final product water, and if trace concentrations are present, they must be below the maximum contaminant level. Other considerations includes that the draw solute should be water-soluble, solid at ambient temperature and pressure, it can be safely handled, besides being cost effective to ensure economic viability of the forward osmosis process.

Achilli et al. (2010) developed a protocol for the selection of optimal draw solutions for forward osmosis applications. It included a desktop screening process and laboratory and modelling analyses. The desktop screening process resulted in 14 draw solutions, which were evaluated for water flux and reverse salt diffusion. Draw solution reconcentration was evaluated using reverse osmosis system design software namely IMS Design (M/s Hydranautics, Oceanside, CA) and Reverse Osmosis System Analysis (M/s Dow Filmtec, Midland, MI) based on water flux, reverse salt diffusion, reverse osmosis permeate concentration and replenishment cost. Analysis of experimental data and model results, combined with consideration of the costs associated with the forward and reverse osmosis processes showed that a small group of seven draw solutions were the most suitable. The different characteristics of these draw solutions highlighted the importance of considering the specific forward osmosis application and membrane types being used prior to selecting the most appropriate draw solution. It was proven that internal concentration polarization is strongly dependent on the diffusion coefficient of the draw solution. The protocol developed can be used for draw solution selection for the new forward osmosis membranes and draw solutions applied to new forward osmosis applications. Kim et al. (2012) also proposed a systematic approach to find designing commercially viable and

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optimal draw solute for viable forward osmosis desalination process. Selection of a draw solute and its concentration mainly decides the water flux through forward osmosis membrane, as well as the energy requirement for draw solute recovery, i.e. the economy of forward osmosis desalination.

More often than not, concentrated solution of NaCl is used as a draw solution due to its high solubility, non-toxic nature, and can be concentrated using conventional desalination techniques such as reverse osmosis or distillation without risk of scaling. Other chemicals such as CaCl<sub>2</sub>, KHCO<sub>3</sub>, MgCl<sub>2</sub>, MgSO<sub>4</sub>, NaHCO<sub>3</sub>, CaCl<sub>2</sub> and MgCl<sub>2</sub> have also been suggested and tested as draw solutes.

Gray et al. (2006) demonstrated that a molar concentration of sodium chloride, dextrose or sucrose used as an osmotic agent solution towards active layer of membrane and water on the other side (mode I) did not have significant effect on transmembrane flux (Figure 3a) due to insignificant internal and external concentration polarizations. In this situation, molecular weight of solute had a negligible impact on performance and flux varied with linearly with osmotic pressure. Whereas, reversing the membrane orientation (mode II) resulted in reduced flux due to substantial internal concentration polarization (Figure 3b). Sodium chloride generated higher flux than dextrose at the same bulk osmotic pressure, which in turn generated more flux than sucrose. It may be attributed to the fact that larger molecular weight solutes could not diffuse as quickly through the porous support of the membrane and dilutive internal concentration polarization would be more severe, resulting in lower flux. Petrotos et al. (1998, 1999) investigated the concentration of tomato juice with forward osmosis using CaCl<sub>2</sub>, Ca(NO<sub>3</sub>)<sub>2</sub>, and

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NaCl. McCutcheon *et al.* (2005, 2006) reported a method for sea water desalination using athermolytic draw solution based on ammonia and carbon dioxide.

For the first time, Ling *et al.* (2010) used highly water-soluble magnetic nanoparticles as novel draw solutes in forward osmosis. The nanoparticles capped with polyacrylic acid yielded the highest driving force and subsequently highest water flux. The used magnetic nanoparticles were captured by the magnetic field and recycled back into the stream as draw solutes in the forward osmosis process. Ling and Chung (2011) developed a potentially sustainable integrated forward osmosis-ultrafiltration system for water reuse and desalination with the aid of super hydrophilic nanoparticles as draw solutes. During forward osmosis, water was transferred from salt solution to the draw solution (containing super hydrophilic nanoparticles), which was regenerated from draw solution using ultrafiltration.

Li *et al.* (2011b) demonstrated that new composite polymer hydrogel particles with light-absorbing carbon particles incorporated within them can be used as draw agents, which were able to draw pure water through forward osmosis membranes, and the water could be recovered by pressure or heating, or a combination of both. The incorporation of light-absorbing particles led to natural, enhanced swelling, heating and dewatering of the composites compared to pure hydrogels during irradiation with light. With an increase in loadings of carbon particles, the water recovery rates from the swollen composite hydrogels were found to be greatly enhanced.

Kim *et al.* (2011) and Liu *et al.* (2011) also utilized naturally non-toxic magnetoferritin as a draw solute for drawing water in forward osmosis process. The drinking water separated from draw solution without use of any intensive energy such as hydraulic pressure or heat. Also, this

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process was not afflicted by the commonly observed problem of reverse salt diffusion. All these characteristics of this novel draw solution separation system make forward osmosis to be an ecosustainable process.

#### 4. Membranes for forward osmosis

The most ideal membrane best suited for forward osmosis must allow higher water flux, high solute rejection, and thin membrane with minimum porosity, high hydrophilicity, reduced membrane fouling and high mechanical strength. The selection of membrane is one of the most critical aspects to be considered in designing forward osmosis process. Any dense, non-porous, selectively permeable material commonly used for reverse osmosis can be used as a membrane for forward osmosis. But, it was not found to be the most suitable due to thick support layer leading to the occurrence of concentration polarization within the membrane support structure and may require very large osmotic driving forces to sustain adequate water flux (McCutcheon et al., 2005; Dova et al., 2007a, b; McGinnis et al., 2007; Gray et al., 2006). A number of researchers studied the feasibility of using available membrane materials such as bladders of pigs, cattle, and fish, collodion (nitrocellulose), rubber, porcelain and goldbeaters' skin (Cath et al., 2006, Tang and Ng, 2008).

In case of reverse osmosis process, the concentration polarization or membrane fouling take place only on the feed side of the membrane (porous support layer backing the membrane contributes insignificant resistance), however, during forward osmosis process, influence on mass transfer will occur on both feed and permeate sides of the membrane (Gray *et al.*, 2006; McCutcheon *et al.*, 2006). Loeb *et al.* (1997) considered to remove the support fabric layer

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bonded to the porous support layer of asymmetric membranes of the Loeb-Sourirajan type of membrane and used these membranes in osmosis process, which were affected due to the membrane orientation. Also, McCutcheon and Elimelech (2008) studied the influence of membrane support layer hydrophobicity on water flux in osmotically driven membrane processes and demonstrated that membrane support layer significantly influences the water flux in osmotically driven membrane process.

McGinnis and Elimelech (2007) demonstrated that the water permeability (as obtained from the slope of Figure 4a) of the cellulose triacetate membrane was slightly lower than the AG and CE membranes (polyamide thin film composite membrane and cellulose acetate asymmetric membrane both have thick fabric backing layers), however, there was a great difference among the osmotic fluxes (Figure 4b). The water permeability differences between the membranes did not account for the vast differences in water flux for the experiments described in Figure 4b. Since permeability does not account for this flux difference, the driving force must somehow be affected by certain other membrane characteristics. It is expected that these thick fabric and porous support layers in both the membranes contribute to the development of internal concentration polarization and, therefore, dramatically reduce the effective driving force and resulting water flux.

Later, Hydration Technologies Inc., USA developed a special membrane for forward osmosis made of cellulose triacetate (thickness less than 50 µm, Figure 5a and 5b), which lacked a thick support layer, but it was embedded in a polyester mesh to provide mechanical support (McCutcheon *et al.*, 2005). A scanning electron microscope images of the cross section of cellulose acetate, polyamide composite and cellulose triacetate membrane is presented in Figure

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6, Ng *et al.*, 2006). This membrane was quite different from standard reverse osmosis membranes. Reverse osmosis membranes typically consisted of a very thin active layer (less than 10 μm) and a thick porous support layer. A comparison of the performance of cellulosic membrane from Hydration Technologies Inc. (designated as CA) and reverse osmosis membrane from General Electric Inc. (Osmonics) (designated as AG) indicated that the water fluxes achieved for the AG membrane were only 7.7% of the CA membrane fluxes when deionized water was used as feed (Figure 7, Garcia-Castello *et al.*, 2009). Similarly, McCutcheon *et al.* (2005) have also indicted that the flux obtained using AG membrane was around 5% of the CA membrane, when NaCl and ammonium–carbon dioxide solutions were used as feed and osmotic agent solutions, respectively.

Forward osmosis membrane can also be made up as hydration bag, which can be used for the recovery of water (Figure 8). It is a double lined bag. The internal bag is made of a forward osmosis membrane and is filled with draw solution (e.g. flavored sucrose) and the external bag is a sealed plastic bag. Upon immersion of the bag in an aqueous solution, water diffuses into the bag due to the osmotic pressure difference and slowly dilutes the draw solution. The concept of hydration bag was developed for military, recreational, and emergency relief situations where reliable drinking water was scarce or not available. Hydration bag is one of the few commercial applications of forward osmosis (Cath *et al.*, 2006).

A novel dual-layer hollow fiber nanofiltration membrane for forward osmosis suitable for protein enrichment/concentration and water production using coextrusion technology was developed. It consisted of an ultra-thin selective skin (around 10 µm), fully open-cell water channels underneath and a microporous sponge like support structure, which could achieve a

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high throughput and purity for lysozyme enrichment with less protein fouling and high salt rejection (Yang *et al.*, 2009a, b).

With a view to reduce the internal concentration polarization, Wang *et al.* (2010a) designed a cellulose acetate forward osmosis membrane comprising of a highly porous sublayer sandwiched between two selective skin layers by using phase inversion technique. The double skin layers of the membrane prevented the salt and other solutes in draw solution from penetrating into the membrane porous support. Moreover, it resulted in higher water flux and lower salt transport. Zhang *et al.* (2010) also developed a double dense membrane structure using phase inversion of cellulose acetate that produced low salt leakage and resulted in less internal concentration polarization in forward osmosis process. Later, Wang *et al.* (2010b) fabricated polyethersulfone thin-film composite hollow fiber membrane for forward osmosis by a dry-jet wet spinning process with an ultra-thin reverse osmosis like skin layer (300–600 nm) on either surface of a porous hollow fiber substrate by interfacial polymerization. The active layers presented excellent intrinsic separation properties with a hydrophilic rejection layer and good mechanical strength.

A high-flux and high-rejection forward osmosis membranes for water reuse and seawater desalination was developed by fabricating polybenzimidazole nanofiltration hollow fiber membranes with thin wall and desired pore size by non-solvent induced phase inversion, which was chemically modified by cross-linking with p-xylylene dichloride (Wang *et al.*, 2009). The cross-linking finely tuned the mean pore size and enhanced the salt selectivity. Further, Su *et al.* (2010) developed cellulose acetate nanofiltration hollow fiber membranes suitable for forward osmosis processes by subjecting it to a two-step heat-treatment (60 °C, 60 min and 95 °C, 20

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min), which effectively shrunk the membrane pores on the membrane surface with a denser outer skin layer (mean pore radius reduced from 0.63 to 0.30 nm). The resultant fiber has a high rejection to NaCl and MgCl<sub>2</sub> and low pure water permeability. Chou *et al.* (2010) developed forward osmosis hollow fiber membrane fabricated by dry-jet wet spinning process. The outer UF skin was made by increasing the air gap in the spinning process and inner RO skin layer was made by interfacial polymerization using m-phenylenediamine aqueous solution and trimesoyl chloride hexane solution. The developed membrane presented excellent intrinsic separation properties with a water flux of 42.6 L.m<sup>-2</sup>.h<sup>-1</sup>. Yip *et al.* (2010) made a high performance thin-film composite membrane consisting of a selective polyamide active layer formed by interfacial polymerization on top of a polysulfone support layer fabricated by phase separation onto a thin (40 µm) polyester nonwoven fabric.

Jia *et al.* (2010) demonstrated the suitability of the carbon nanotube membranes for seawater desalination using forward osmosis. The membrane could achieve not only the optimum salt rejection property but also the largest water flux, which broke the limit of trade-off effect between selectivity and permeability existing in the traditional liquid separation membrane. The anti-fouling ability and good mechanical strength rendered it more suitable for forward osmosis. According to a rough estimation, the carbon nanotube membranes can achieve a higher water flux far in excess of the existing commercial FO membranes.

Qiu et al. (2011) successfully fabricated forward osmosis membranes using layer-by-layer assembly of polyallylamine hydrochloride and polysodium 4-styrene-sulfonate on a porous polyacrylonitrile substrate. The chemical cross-linking of layer-by-layer polyelectrolyte layers was performed with glutaraldehyde. The cross-linked and non cross-linked membranes both the

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membranes had relatively high water permeability. But, the cross-linked membranes showed better MgCl<sub>2</sub> rejection, which clearly demonstrated the potential of layer-by-layer membranes for high flux forward osmosis applications. Later, Qiu *et al.* (2012) fabricated high performance flat sheet forward osmosis membranes using polyamide-imide materials via phase inversion followed by polyelectrolyte polyethyleneimine post-treatment to form a nanofiltration-like rejection layer with positive charges. Polyamide-imide micro-porous substrate was embedded with a woven fabric, the enhanced mechanical strength of the membrane made it possible to reduce the thickness of the substrate to 55 μm and the resultant membranes can reach a water flux of 29.65 and 19.2 Lm<sup>-2</sup>h<sup>-1</sup> in case when the active layer facing draw or feed solutions, respectively.

#### 5. Applications of forward osmosis in Food processing

A very wide range of applications have been studied for forward osmosis membrane processes. Since the forward osmosis is an osmotic pressure driven ambient temperature process, it promotes high retention of sensory (e.g. taste, aroma, color) and nutritional (e.g. vitamin) quality. High rejection and low membrane fouling compared to pressure-driven membrane processes like reverse osmosis are the additional advantages. Several patents are available for the development of forward osmosis membranes, devices and methods for the concentration of liquid foods (Foreman *et al.*, 1977; Liu *et al.*, 2009). The work carried out by various researchers on forward osmosis in the area of food processing has been summarized in Table 1.

#### 5.1 Grape juice

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Popper *et al.* (1966) were the first to report the concentration of grape juice using forward osmosis process involving a plate and frame filter press with a reverse osmosis type of membrane made of flat sheet cellulose acetate membranes. Sodium chloride was used as a draw solution. With moderate stirring, the grape juice was concentrated from 16 to 60° Brix with a flux of 2.5 L.m<sup>-2</sup> h<sup>-1</sup>.

#### 5.2 Tomato juice

Petrotos et al. (1998) have investigated the use forward osmosis in tubular configuration for the concentration of tomato juice. The tomato juice was circulated through the systems inside the bore of the tubes and the tube arrangement and connection was in series with the assistance of a U-bend element. The osmotic medium was recirculated each time outside the tubes in the shroud. The overall arrangement of the forward osmosis experimental rig is shown in Figure 9. The membrane module consisted of an external stainless steel shroud accommodating internally a set of two identical reverse osmosis membrane tubes having no support lengthwise and properly sealed at their ends. The process performance was measured in terms of water permeation flux and its response to changes of the process parameters was experimentally assessed and established. A comparison was carried out by using different draw solution such as sodium chloride, calcium chloride, calcium nitrate, glucose, sucrose or polyethylene glycol 400. Sodium chloride was found to be the most effective osmotic agent among all the draw solutions. The correlation coefficient between driving force  $(\Delta \pi - \Delta P)$  and flux values for different osmotic solutes were found to be very low (Table 2), which indicated that the performance of the process of forward osmosis is dependent on the diffusion of water from the membrane active layer through the membrane backing material and osmotic medium polarized layer, which is in turn

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dependent on the physical properties of the used osmotic solution. At the same time flux values were found to be inversely proportional to the square root of the viscosity (Figure 10a), which signified the importance of using a low viscosity osmotic medium in forward osmosis. Osmotic flux was found to traverse exponential trend with the thickness of membrane backing material with the active membrane layer being the same. The reduction in the thickness resulted in increase in osmotic flux (Figure 10b). Petrotos *et al.* (1998) have also reported that in forward osmosis process the increase in temperature resulted in an increase in transmembrane flux in the case of the concentration of tomato juice (Figure 10c). The increase of temperature lowers the viscosity and increases the diffusion coefficients of the fluids involved in the process, thus positively affecting the permeation flux.

Petrotos *et al.* (1999) studied the effect of clarifying tomato juice on the rate of forward osmosis concentration. The different clarification pretreatments such as filtration of juice by screen (35 mm mesh) or ultrafiltration (molecular weight cut-off 200, 100 or 25 kDa) was compared. It was observed that the use of ultrafiltration pretreatment juice improved the performance of the forward osmosis concentration instead of carrying the concentration of tomato juice as such. The decrease in molecular weight cut off from 200 to 100 kDa resulted in enhanced flux rates whereas further decrease in molecular weight cut off was not found to have any significant effect of the forward osmosis performance (Figure 11). This study demonstrated a great potential in using a combined low temperature and pressure ultrafiltration and forward osmosis process to produce tomato concentrates. Petrotos *et al.* (2010) developed a forward osmosis apparatus with a flat configuration (Figure 12). The block diagram of the forward osmosis is presented in Figure 13a. The main part of the apparatus was an osmotic module. The

module was in the shape of a flat configuration consisting of two parts (Figure 13a and 13b) 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 draw solution in the two respective sides of the membrane and enabled the osmotic transfer of pure water from the juice to the osmotic medium side. For re-concentration of the post-process diluted osmotic NaCl brines, electrodialysis was proposed as a viable alternative to the commonly used evaporative process, which is an established methodology in the chemical industry for concentrating brines up to the saturation and is far more economical than evaporation. A combination of forward osmosis—electrodialysis process operated at ambient temperature and low pressure was demonstrated to be suitable for the production of tomato concentrate.

#### 5.3 Pineapple juice

Forward osmosis was employed for the concentration of pineapple juice by adopting a combination of aqueous solution of sucrose (0–40%, w/w) and sodium chloride (0–26%, w/w, Babu *et al.*, 2006), which could overcome the drawback of using sucrose (low flux) or sodium chloride (salt migration) individually as an osmotic agents during forward osmosis process. The increase in sodium chloride concentration (0 to 16%, w/w) at a fixed concentration of sucrose (30%, w/w) resulted in increase in transmembrane flux, however, it led to increased sodium chloride migration up to 1.28% to the feed side (Figure 14a). Similarly, the increase in sucrose concentration (0 to 40%, w/w), to a fixed sodium chloride solution (12%, w/w) resulted in an increased transmembrane flux and decreased sodium chloride transfer to the feed side (from 1.87).

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to 0.58%, Figure 14b). The overall mass transfer coefficient (K) was found to decrease with increase in draw solution concentration (Figure 14c, d). Since in all the cases the concentration of pineapple juice and hydrodynamic flow conditions are same on both sides (feed side and osmotic agent solution side), the reduction was due to change in physical properties of draw solutions, which affect the draw solution side film coefficient and in turn diffusion through the membrane. The pineapple juice was concentrated up to a total soluble solids content of 60 °Brix. The effect of forward osmosis process on physico-chemical characteristics of pineapple juice indicated that the ascorbic acid content was well preserved in the pineapple juice concentrate (Babu *et al.*, 2006).

#### 5.4 Anthocyanin extract

Rodriguez-Saona *et al.* (2001) developed a process for the concentration of red radish (anthocyanin) extract using forward osmosis as a potential natural red colorant. The red radish juice was extracted using abrasive peeler. The juice and disrupted tissues were mechanically pressed to obtain juice, which was blanched, cooled, pH adjusted, filtered and concentrated in centrifugal film evaporator (Centitherm) before it was subjected to forward osmosis (Figure 15). The use of abrasive peeler to extract the pigments was very efficient in giving low concentration (~1 °Brix) with high anthocyanin recoveries (> 90%) and reduced levels of aroma compounds. Concentration of the extracts gave colorants with desirable color attributes and minimal pigment degradation. Radish concentrates had color characteristics close to FD&C Red #40. The extract was concentrated up to 30° Brix and contained anthocyanin levels of 800mg/100mL. The

combination of thermal evaporation and forward osmosis was found to considerably reduce the levels of undesirable radish aroma compounds.

Nayak and Rastogi (2010a, b) demonstrated that the concentration of anthocyanin extract from Garcinia indica Choisy using forward osmosis has several advantages over the thermal concentration in terms of higher stability, lower browning index and less conversion of hydroxy citric acid (HCA) to its lactone form. Anthocyanin was concentrated from 49 mg/l to 2.69 g/l (54-fold increase) using forward osmosis asymmetric membrane consisting of active skin layer of cellulose triacetate embedded in a nylon mesh in a flat membrane module having a membrane area of 1.14 x 10<sup>-2</sup> m<sup>2</sup>. The authors studied the effect of various process parameters on transmembrane flux and anthocyanin content. The flux in case of anthocyanin extract was found to increase in both the modes I and II with an increase in osmotic agent (NaCl) concentration. The flux value in mode II was found to be higher in comparison to mode I due to the negligible concentration polarization. The increase in feed flow rate from 50 to 125 ml/min resulted in increase in flux in both the modes I and II (Figure 16a). Similarly, flux was found to increase in both the modes I and II with an increase in osmotic agent flow rate (Figure 16b). At the same time, the anthocyanin concentration was found to increase for all the cases with an increase in transmembrane flux. Also, Nayak and Rastogi, (2010a) indicated that flux values and anthocyanin content in modes I and II were found to increase with an increase in feed temperature (Figure 16c). The variation of the increase in transmembrane flux with temperature can be explained by Wilke-Chang equation (Treybal, 1982), according to which, the diffusion coefficient is proportional to the absolute temperature divided by the viscosity of the solvent. The increase in temperature reduces the viscosity of solution and increases the diffusion

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coefficients, which results in increase in transmembrane flux (Petrotos *et al.*, 1998, McCutcheon and Elimelech, 2006).

#### 5.5 Sugar solution

The effect of process parameters on performance of a pilot scale forward osmotic (membrane) concentration unit was studied by Dova et al. (2007a). The unit consisted of a flat geometry membrane module with a contacting area of 0.09 m<sup>2</sup> (1.0 ft<sup>2</sup>). Flat, square, reverse osmosis-type membrane sheets with varying characteristics were used with the osmotic module (cell). The deionized water and low concentration sucrose or glucose solutions were utilized as feed solutions to study (and model) the impact of experimental parameters on process performance. Solution of sodium chloride was used as a draw solution. Osmotic flux increased linearly with the osmotic medium concentration (Figure 17a), however, the mass transfer coefficient decreased by 75% with increased osmotic medium concentration (Figure 17b), which indicated the need to reduce mass transfer resistance on the brine side by improving the transport properties of the backing material by making as thinner as possible as well as contact conditions. As feed concentration was increased from 0.1 to 0.6, the flux was found to decrease for both 10 and 23% brine solutions (Figure 17c). Despite the significant difference in the osmotic pressures, the values were not significantly different for highest feed concentration (0.6 M). The proportional increase in flux was not observed due to higher boundary layer resistance on the feed side (due to increased viscosity and concentration polarization phenomena) and decreased osmotic pressure differential. However, overall mass transfer coefficient was found to be lower for 23% NaCl solution as compared to 10% NaCl solution due to higher concentration

polarization (Figure 17d). Dova *et al.* (2007b) developed a generalized model for the forward osmotic concentration process.

Garcia-Castello *et al.* (2009) demonstrated that the sugar solution can be concentrated using sodium chloride as a draw solution. This study demonstrated that forward osmosis is a viable alternative membrane process for concentration for sucrose solution. A concentration factor of 5.7 was achieved by forward osmosis with a starting sucrose concentration of 0.29 M. Water fluxes were reported to be lower than those commonly obtained in reverse osmosis, which was a consequence of the significantly higher concentration factors in conjunction with internal concentration polarization. The dependence of water flux on the osmotic pressure difference between the draw solution and feed solution bulk osmotic pressures for cellulosic (CA) and reverse osmosis (AG) membrane is shown in Figure 18a, which indicated that higher transmembrane bulk osmotic pressure differences generated by the 4.0 M draw solution yielded similar water fluxes as those driven by the 2.0 M draw solution. These differences were due to the more severe internal concentration polarization phenomena associated with more concentrated draw solutions. The influence of dilutive internal concentration polarization on water flux represented by considering the relationship between water flux and the transmembrane bulk osmotic pressure difference normalized by the feed side osmotic pressure indicated that at low normalized driving forces (Figure 18b), water flux strongly increased with increasing osmotic driving force. However, at higher normalized driving force, the increase in water flux diminished substantially due to increasing severity of internal concentration polarization. It was therefore concluded that the use of most dilute draw solution possible in

order to obtain the required concentration factor of the sucrose solution would be more desirable (Garcia-Castello *et al.*, 2009).

#### 5.6 Model food system

In order to evaluate the effect of molecular weight of feed solution on transmembrane flux, the model systems containing different molecular weight sugars (i.e. 0.15 M dextrose, molecular weight 180 g/mol or 0.15 M sucrose, molecular weight 342.30 g/mol) were used as feed solutions (Nayak et al., 2011). In case of mode I, the transmembrane flux was found to be higher when 0.15 M solution of dextrose was taken as a feed solution in comparison to the same concentration of sucrose solution (Figure 19a) because of the higher penetration power in loosely bound support layer of dextrose in comparison to sucrose due to its lower molecular weight resulting in higher internal concentration polarization. The higher flux in case of mode II was due to negligible external and internal concentration polarization towards feed side and the internal concentration polarization was almost equal for both the solutions towards the draw solution side (Figure 19b, Nayak et al., 2011). In case of mode I, the presence of pectin along with the sucrose and dextrose resulted in further decrease in flux due to significant external concentration polarization on the support layer because of its high molecular weight. The mixture of sugars (0.15 M dextrose and 0.15 M sucrose) plus 0.1% pectin resulted in lowest flux because of increased internal concentration polarization due to increased effective concentration of sugars in addition to the presence of external concentration polarization (Figure 19c). In case of mode II, the fluxes were also lower (Figure 19d) than the corresponding fluxes for feed without pectin as shown in Figure 19b due to the enhanced external concentration polarization

because of pectin. The flux in case of mixture of sugars with pectin was still lower due to higher effective concentration on feed side thereby reducing the effective driving force (Figure 19d, Nayak *et al.*, 2011).

#### 5.7 Beetroot juice

The beetroot juice was concentrated by placing active layer towards feed and draw solution towards the support layer of the forward osmosis membrane. The variation in the flux and °Brix indicated that the beetroot juice was concentrated in 2 cycles (6 h of continuous concentration was referred as 1 cycle) and the betalains content of the juice was found to increase from 50.9 mg/L to 2.9 g/L (57.1 fold) and total soluble solids was found to increase from 2.3 to 52 °Brix (22.6 fold, Figure 20a). The fluxes were found to decrease in the each cycle, but these were found to increase in the beginning of next cycle due to washing of the membrane with distilled water after each cycle. The pineapple juice was concentrated in 3 cycles and the total soluble solids was found to increase from 4.4 to 54 °Brix (12 fold, Figure 20b). The anthocyanin from the grape juice was concentrated in 4 cycles from 104.8 to 715.6 mg/L (6.8 fold) and total soluble solids from 4.4 to 54 °Brix (12.3 fold, Figure 20c).

#### 5.8 Red raspberry juice

The composition and sensory quality of red raspberry juice concentrated by forward osmosis with the juice concentrated by conventional evaporation was compared (Wrolstad *et al.*, 1993). Raspberry juice was concentrated up to 45 °Brix by evaporation as well as forward osmosis. Concentration by either process resulted in small anthocyanin pigment losses and

formation of fumaric acid and small increases of polymerized pigment. The aroma and flavor of the experimental samples and nine commercial concentrates were evaluated in a raspberry drink formulation by a trained descriptive flavor panel. Principal component analysis revealed no significant flavor differences between the single-strength juice, the concentrates and three of the commercial samples. The study suggested that the forward osmosis concentrate is of premium quality, compared to that obtained by conventional evaporation. The osmotic concentrates were clustered closer to single strength juice in red raspberry in terms of flavor and aroma than the evaporation-concentrated sample. There were minor compositional differences between the osmotically concentrated and evaporation concentrated sample.

#### 5.9 Orange peel press liquor

Garcia-Castello and McCutcheon (2011) evaluated the use of forward osmosis dewatering process as an alternative for dewatering orange peel press liquor. The membrane unit was custom built with channels (77 mm long × 26 mm wide × 3 mm deep) on both sides of the membrane. The press liquor was concentrated by removing water by using sodium chloride as a draw solution. Concentration factors up to 3.7 resulted when 4.0 M NaCl draw solution was used. The feed solutions with no pectin (S#1 and S#3) experienced the less flux decline as compared to the feed solutions containing pectin (S#2, S#4, and S#5), which indicated primary role of pectin in membrane fouling during press liquor dewatering. The feed solution containing pectin, citric acid and no calcium chloride (S#2) resulted in slightly less flux decline. However, the feed solution containing calcium chloride and pectin without citric acid (S#5) resulted in the most severe initial flux decline. The feed solution containing calcium chloride, pectin and citric

acid (S#4) the fouling was only slightly decreased. It has been shown that calcium ions preferentially formed complex with citric acid. Therefore, when both constituents were present, pectin and citric acid compete to complex with calcium ions. When citric acid was removed, all of the calcium ions were free to complex with the pectin instead of causing more severe fouling (Figure 21a). This fouling layer led to cake enhanced concentration polarization, which masked the increased driving force difference created by the higher draw solution concentration. After fouling, the flux for feed solutions containing pectin (S#2, S#4, and S#5) was the same for both the 2.0 and 4.0 M draw solution (Figure 21b).

#### 5.10 Protein solution

Yang *et al.* (2009a, b) demonstrated that dual-layer hollow fiber nanofiltration membranes can be successfully used in the forward osmosis for the enrichment and concentration of pharmaceutical products without denaturing the component of interests. A high throughput for lysozyme enrichment and less protein fouling in addition to the high divalent salt rejection ensured the enriched lysozyme product with high purity and without change and denaturing. The lysozyme concentration and enrichment factor was higher when feed solution was towards the support layer as compare to the situation when feed solution was towards active layer due to more significant internal concentration polarization occurred in the latter situation in which the inner porous structure of asymmetric membrane seriously decreased the net driving force and led to a reduced water permeation flux (Figure 22). A small membrane surface area (70 cm<sup>2</sup>) enriched lysozyme and concentrated it by a factor of 1.85 using 600 mL 3.12 M MgCl<sub>2</sub> as the draw solution in 3 h.

Wang *et al.* (2011) developed an integrated forward osmosis-membrane distillation hybrid system for the concentration of protein solutions, specifically a bovine serum albumin (BSA) solution (Figure 23). A concentrated sodium chloride solution was employed as the draw solution (0.5M) to dehydrate proteins in forward osmosis, while distillate water was a by-product during the re-concentration of diluted sodium chloride draw solution in membrane distillation. The hybrid system was reported to be a promising method for the concentration of pharmaceuticals/protein solutions. The BSA and sodium chloride concentrations in the feed protein solution were found to increase with time (Figure 24a and 24b). A reasonably stable forward osmosis water transfer rate could be maintained during the entire course of experiment, and it matched more accurately to that of membrane distillation at 54°C resulting in no significant change in the concentration of draw solution (Figure 24c and 24d). It was also demonstrated by the authors that BSA does not exert significant osmotic pressure on solution and therefore the trans-membrane osmotic pressure gradient and thus water flux does not depend greatly on the BSA feed concentration.

It is evident that the forward osmosis process has enormous advantages over conventional concentration methods such as thermal evaporation and pressure driven membrane processes for concentration of liquid foods. Low energy usage, low operating temperatures and pressures as well as high product concentration are the main advantages of this process.

#### 5.11 Desalination

Over the years, purified water standards have become more stringent. However, membranes have risen to the challenge and continue to perform efficiently and effectively and

forward osmosis membranes can be used for the desalination of water. Forward osmosis is a process that may be able to desalinate saline water sources at a notably reduced cost.

Frank (1972) described a method of forward osmosis using a solution of aluminum sulfate as an osmotic agent, which on treatment with calcium hydroxide resulted in precipitate of aluminum hydroxide and calcium sulfate, which was removed by standard methods leaving the fresh product water. Kravath and Davis (1975) described a process of seawater desalination by forward osmosis across a cellulose acetate membrane using glucose as draw solute. Upon dilution, the salinity was reduced to a level where ingestion would be possible for short term consumption. The performance of flat sheet cellulose acetate membranes were reported to be poor in terms salt rejection. Stache (1989) used a semi-permeable membrane bag filled with concentrated fructose solution to desalinate small volumes of seawater while simultaneously creating a nutritious drink. Whereas, Yaeli (1992) used a concentrated glucose solution as a draw solution and water was extracted from seawater by osmosis. The diluted glucose solution was fed to an RO unit, where a low pressure RO membrane separated potable water from the sucrose draw solution.

McCutcheon *et al.* (2009) and Chanukya *et al.* (2012) demonstrated a forward osmosis desalination process in which ammonium bicarbonate was used as an osmotic solution to extract water from saline feed water. Forward osmosis using semi-permeable polymeric membranes may be a viable alternative to reverse osmosis as a lower cost and more environmentally friendly desalination technology. Very large osmotic pressures generated by the highly soluble ammonium bicarbonate osmotic solution yielded high water fluxes and resulted in very high feed water recoveries. Ammonium bicarbonate upon moderate heating can be decomposed into

ammonia and carbon dioxide gases leaving the fresh product water. Schematic diagram of the ammonia carbon dioxide forward osmosis desalination process to recover fresh water is presented in Figure 25, McGinnis and Elimelech, 2007). Similarly, Low (2009) studied the performance of forward osmosis with respect to different ammonia-carbon dioxide draw solutions and membranes. The internal concentration polarization resulted in lowering of the flux.

Lampi et al. (2007) integrated forward osmosis process with reverse osmosis process to generate potable water. The forward osmosis was used to generate significant hydraulic pressure used for driving reverse osmosis process, wherein the reverse osmosis process can separate salt from sea water to generate potable water form water with high salt content. Martinetti et al. (2009) compared vacuum-enhanced direct contact membrane distillation (VEDCMD) and forward osmosis for the desalination of brackish water. The water recovery in case of forward osmosis was higher (90%) as compare to VEDCMD (81%). Bamaga et al. (2009) presented a design for an integrated desalination unit consisting of a closed forward osmosis and reverse osmosis process in which reverse osmosis brine was used as the osmotic draw solution and thus the chemical energy stored in the reverse osmosis brine was recovered and utilized by the forward osmosis membrane process. Also, Choi et al. (2009, 2010) investigated the combined systems with forward osmosis and reverse osmosis for seawater desalination. A pilot-scale combined systems with forward osmosis resulted in higher recovery with higher quality and fluxes than conventional reverse osmosis based desalination systems. Similarly, Valladares et al. (2011) focused on the rejection of 13 selected micropollutants by clean or fouled forward osmosis membrane, using Red Sea water as a draw solution. The resulting effluent was then

desalinated at low pressure with a reverse osmosis membrane to produce a high quality permeate. Tan and Ng (2010) proposed a hybrid forward osmosis-nanofiltration process for seawater desalination. The process was found to achieve good quality product water that met the recommended drinking water total dissolved solids guideline (500 mg/L). Also, Zhao et al. (2012) designed a hybrid forward osmosis-nanofiltration system designed for brackish water desalination and also compared with a stand-alone reverse osmosis process. Lay et al. (2010) examined the possible factors for the slower decline of flux in forward osmosis process. The transmission of draw solutes from the draw solution into the feed was found to have significant effect on performance. Yangali et al. (2011) indicated that forward osmosis coupled with low pressure reverse osmosis used for indirect desalination consumed only half of the energy used for high pressure seawater reverse osmosis desalination, and produced good quality water extracted from the impaired feed water. A cost analysis revealed that forward osmosis per se as viable and promising technology. Ling and Chung (2011) developed a potentially sustainable integrated forward osmosis-ultrafiltration system for water reuse. During forward osmosis, water was transferred from salt solution to the draw solution (containing super hydrophilic nanoparticles), which was regenerated from draw solution using ultrafiltration.

#### 6. Concluding remarks

Considering the diversified applications of forward osmosis not only in food processing but also in many other disciplines such as water treatment, waste water treatment, seawater/brackish water desalination, pharmaceutical drug delivery, and electric power production, it may be concluded that the forward osmosis in the most exciting, emanating and

emerging technology for the water removal from liquids due to its novel features, which include no use of solvent for extraction, low energy consumption, higher retention of thermo labile components and attainment of higher concentration. A number of recent patents and publications have demonstrated novel and diverse uses of this technology more and more among academic researchers as well as industries. Although a good number of potential applications of forward osmosis membrane processes have been indicated and investigated for variety of liquid foods, it is still facing some serious and vital challenges. These challenges are issues related with concentration polarization, membrane fouling, reverse solute diffusion, and the need for membrane development and the design of the draw solute. Designing of improved performance forward osmosis membranes in terms of higher flux membranes and most suitable draw solutions may further augment the pace of development of this technology. For example, the advent of double-selective layer membranes improved the membrane design as well as in a way to find ideal draw solution the use of nano-particles, magnetic particles, hydogels were prove to be exceedingly interesting research activities in the area of forward osmosis research. Forward osmosis membranes need to provide high water permeability, high rejection of solutes, substantially reduced internal concentration polarization, high chemical stability, and high mechanical strength. Recovery and regeneration of the draw solution can be construed as the one of the most significant limitations in transforming forward osmosis into a full-scale process in the food industry.

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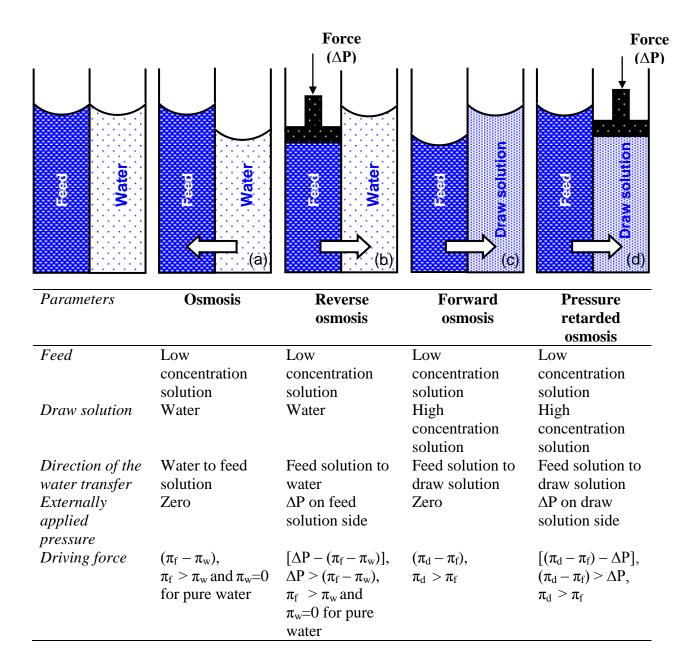
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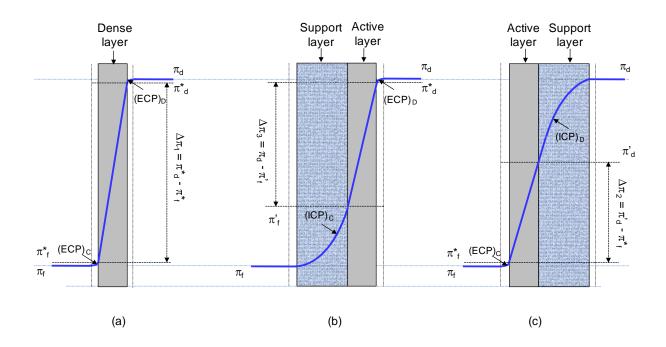
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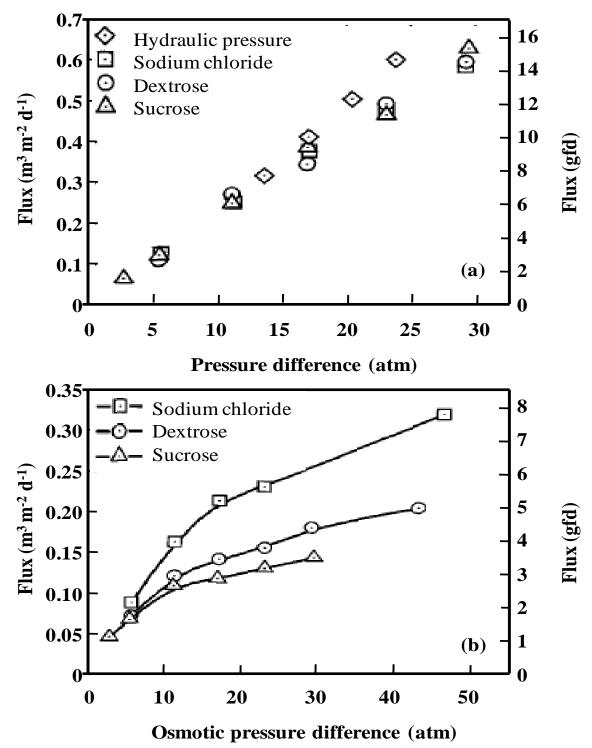
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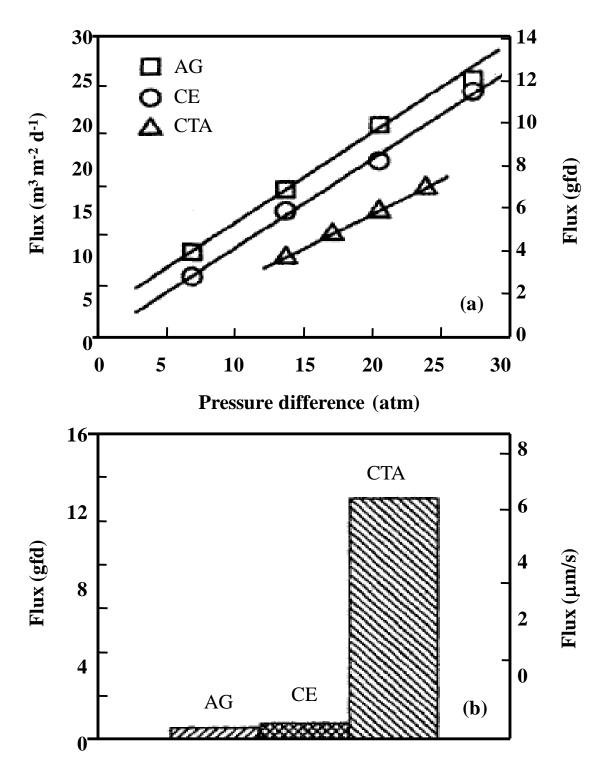
**Fig. 1:** Migration of water during (a) osmosis, (b) reverse osmosis, (c) forward osmosis and (d) pressure retarded osmosis. The arrows indicate the direction of mass transfer.  $\Delta P$  is the hydraulic pressure and  $\pi_f$ ,  $\pi_d$ ,  $\pi_w$  are the osmotic pressures of feed solution, draw solution and water.



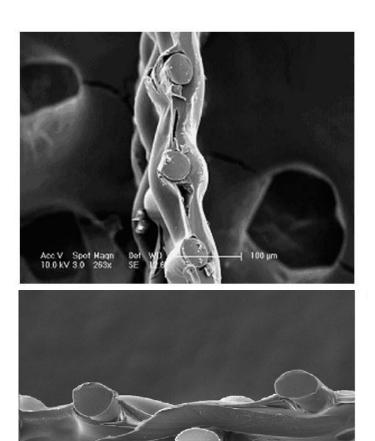
**Figure 2**: Mechanism of forward osmosis indicating water transport (a) with a dense symmetric membrane; with asymmetric membrane (b) feed towards the support layer (mode I) and feed towards active layer (mode II).  $\pi_d$  and  $\pi_f$  are the bulk osmotic pressures of draw and feed solutions, respectively;  $\pi^*_d$  and  $\pi^*_f$  the osmotic pressures on membrane surface of draw and feed solutions, respectively;  $\pi^*_f$  and  $\pi^*_d$  are the osmotic pressures of the feed and draw solutions on the inside of the active layer within the porous support for concentrative internal concentration polarization on feed side and dilutive internal concentration polarization on draw side for mode I and mode II, respectively.  $\Delta \pi_1$ ,  $\Delta \pi_2$  and  $\Delta \pi_3$  are the corresponding effective driving force in (a), (b) and (c) situations, respectively.



**Figure 3:** Water flux as a function of driving force (pressure difference). For the FO runs with the three draw solutions, the osmotic pressure difference is generated by varying the draw solution concentrations. (a) Draw solution was placed against the membrane active layer while the feed (deionized water) in placed against the support layer, (b) Draw solution was placed against the support layer of the membrane and feed (deionized water) in placed against the active layer. (from Grav *et al.*. 1996)

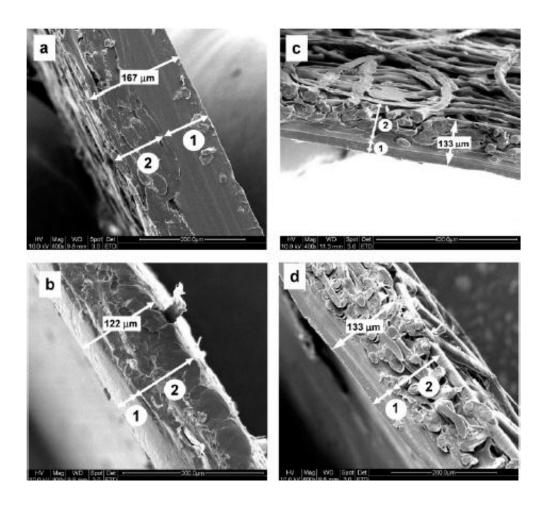


**Figure 4**: (a) Variation of pure water flux with hydraulic pressure, (b) Comparison of water flux in forward osmosis mode for the three membranes tested: AG and CE (reverse osmosis membranes), and CTA (forward osmosis membrane). Experimental conditions were as follows: 6 M ammonium bicarbonate draw solution, 0.5 M sodium chloride feed solution, and temperature of both feed and draw solutions of 50°C. (from McCutcheon *et al.*, 2005)

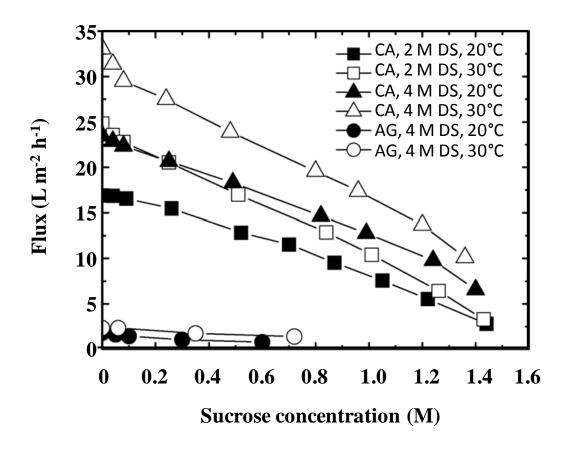


**Figure 5**: SEM images of cross-sections cellulosic forward osmosis membrane (CA). A polyester mesh is embedded within the polymer material for mechanical support. The membrane thickness is less than 50  $\mu$ m. (from McCutcheon *et al.*, 2005; Garcia-Castello *et al.*, 2009)

Acc.V Spot Magn Det WD Exp 10.0 kV 3.0 298x SE 11.6 1



**Figure 6:** SEM images of the cross section of (a) cellulose acetate (CA) membrane; (b) polyamide composite (AD) membrane; (c) FO membrane and (d) FO membrane at higher resolution where '1' is the dense selective layer and '2' is the support layer (from Ng *et al.*, 2006)



**Figure 7:** Effect of temperature, draw solution concentration, and sucrose feed solution concentration on permeate water flux for both the CA and AG membranes. Experimental conditions: feed solution (sucrose) concentration of 0–1.65 M (0-48 °Brix), draw solution concentration of 2 or 4 M NaCl, and cross flow rate for draw and feed solutions of 1 Lmin<sup>-1</sup> (1.67×10–5 m<sup>3</sup> s<sup>-1</sup>). (from Garcia-Castello *et al.*, 2009)

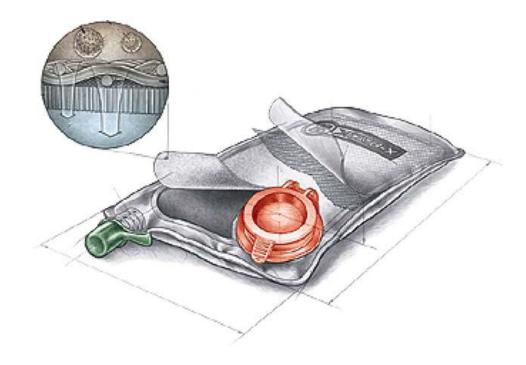
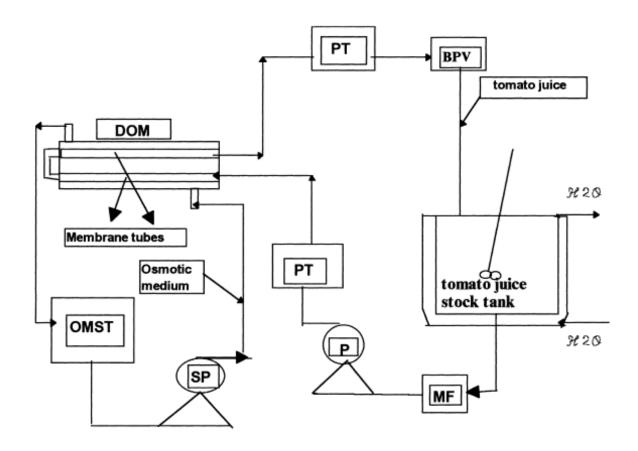
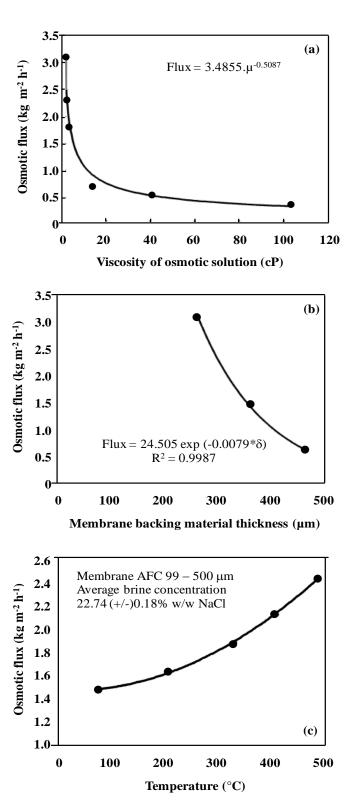


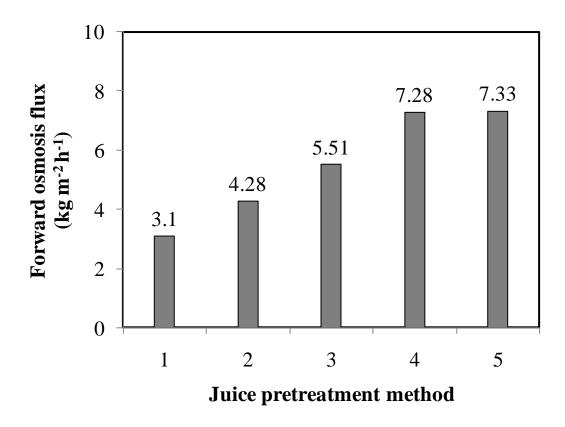
Figure 8: Illustration of water purification hydration bag (from Cath et al., 2006)



**Figure 9:** Typical forward osmosis experimental rig. MF is the magnetic flow meter, P the positive displacement pump, SP the stoke pump, PT the pressure transducer, DOM the forward osmosis module, OMST the osmotic medium stock tank, BPV the back pressure value (from Petrotos *et al.*, 1998)



**Figure 10**: (a) Correlation of the direct osmosis flux with osmotic medium viscosity in direct osmosis concentration of tomato juice; (b) correlation of the tomato juice direct osmosis flux with the membrane backing material thickness; (c) effect of temperature on the tomato juice direct osmosis flux. (from Petrotos *et al.*, 1998)



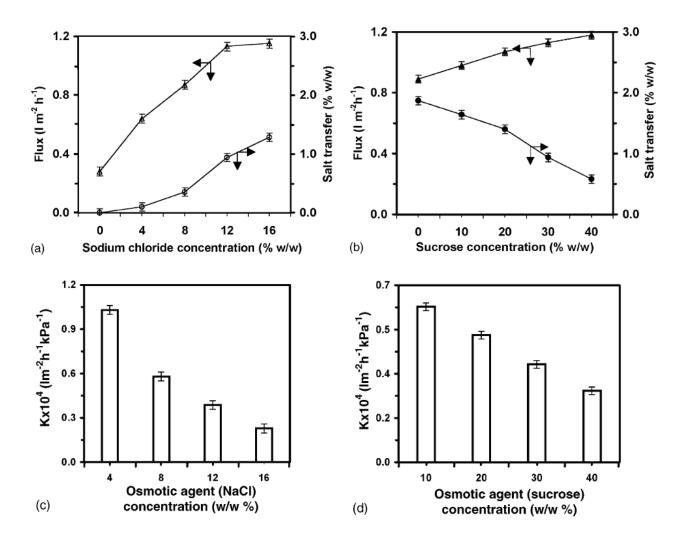
**Figure 11**: The direct osmosis flux value measured with different tomato juice clarifications. Juice pretreatment methods 1, 2, 3, 4 and 5 refer to control, screen with 38 μm mesh, 200, 100 and 25 kDa, respectively (from Petrotos *et al.*, 1999)



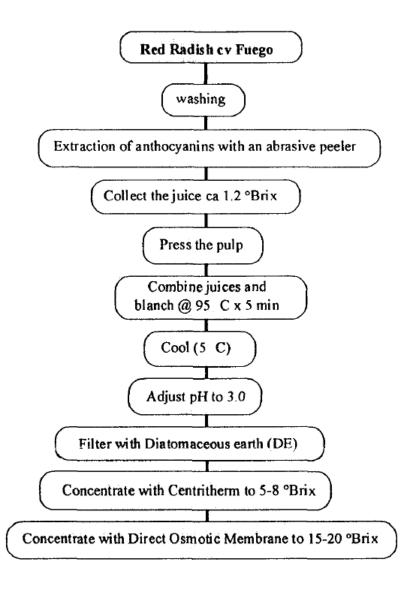
Figure 12: Experimental forward osmotic rig (Dova et al., 2007).

#### ACCEPTED MANUSCRIPT Stirrer Brine stock tank Tomato juice stock tank Pressure Pressure Pressure Pressure Control valve Control valve meter meter Pressure Pressure Control valve Control valve Pressure Osmotic Pressure (a) Preumaticme œll meter meter Pneumaticme mebrane mebrane pump pump \_\_\_\_\_\_ 0 0 0 0 0 0 0 0 0 0 0 0 0 00000000000000 \_\_\_\_\_\_ **(b)**

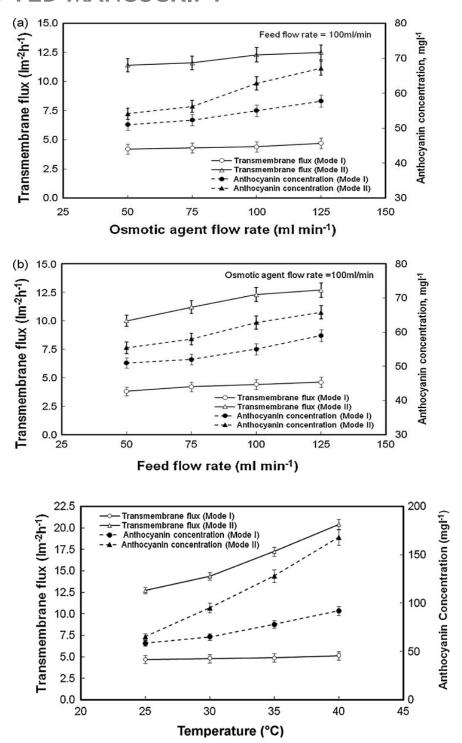
**Figure 13:** (a) Block diagram of experimental set up for the forward osmosis concentration of tomato juice (b) geometry of Part A and B of osmotic cell in flat membrane module configuration Petrotos *et al.* (2010)



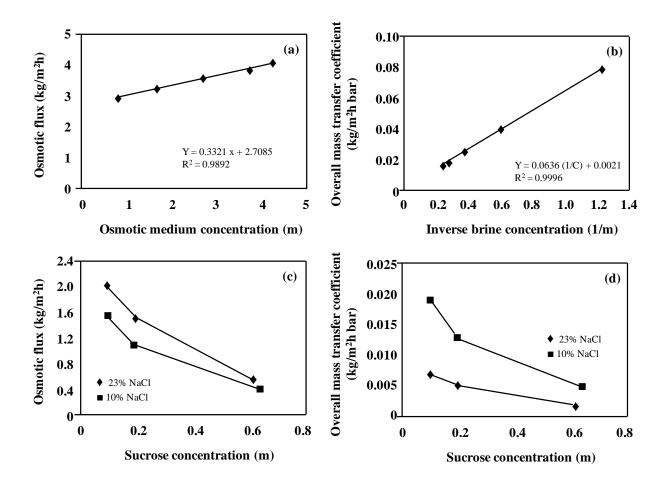
**Figure 14:** Effect of mixed osmotic agent on flux and salt transfer during direct osmosis. (a) addition of sodium chloride (0–16%, w/w) to sucrose (30%, w/w); (b) addition of sucrose (0–40%, w/w) to sodium chloride (12%, w/w). (c, d) Effect of osmotic agent concentration on overall mass transfer coefficient (Babu *et al.*, 2006)



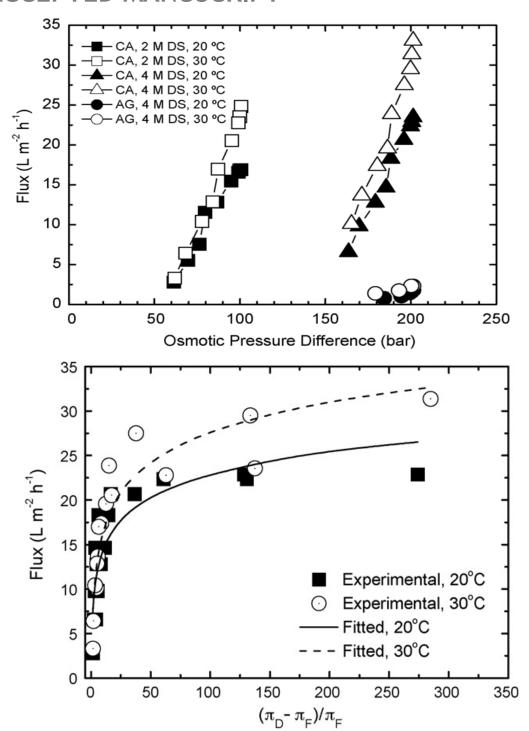
**Figure 15**: Flow chart of the unit operations for the production of radish concentrate extracts (from Rodriguez-Saona *et al.* (2001)



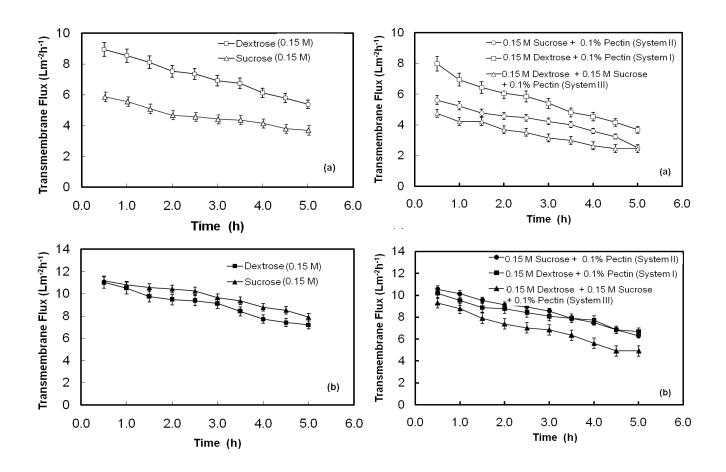
**Figure 16**: Effect of (a) feed, and (b) osmotic agent flow rate on transmembrane flux and anthocyanin concentration; (c) effect of temperature on transmembrane flux and anthocyanin concentration. Modes I and II were referred to the membrane orientation in which feed was towards the support layer and feed was towards active layer, respectively. Osmotic agent concentration was maintained at 6.0 M. (from Nayak and Rastogi, 2010a)



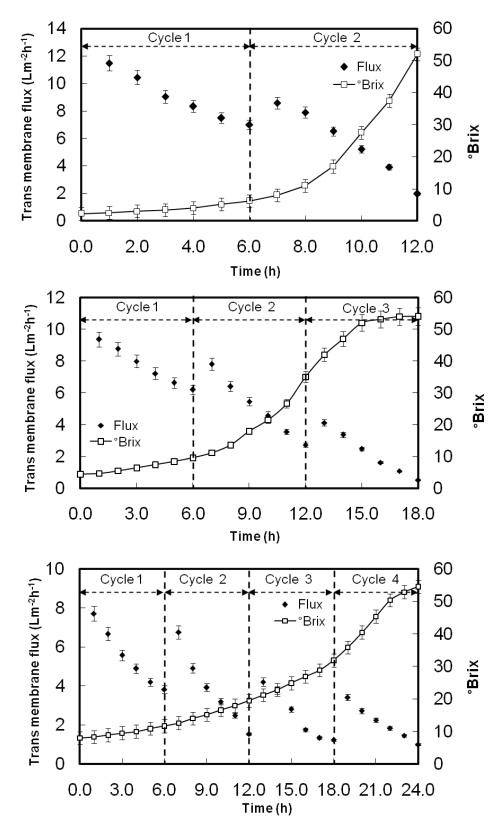
**Figure 17**: The effect of osmotic medium concentration on the forward osmotic flux; (b) impact of osmotic medium concentration on overall mass transfer coefficient (c) impact of feed (sucrose) concentration on osmotic flux (d) impact of feed (glucose) concentration on osmotic flux (from Dova *et al.* (2007a).



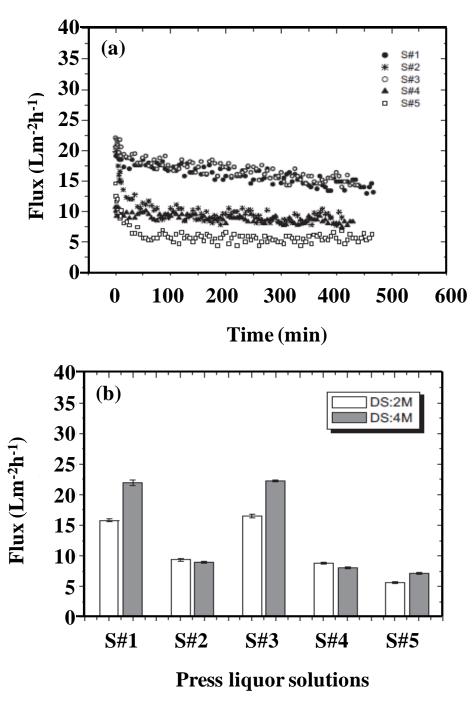
**Figure 18:** (a) Effect of temperature, feed solution concentration, and osmotic pressure driving force on permeate water flux for both the CA and AG membranes; (b) water flux for the CA membranes as a function of the osmotic pressure difference between the draw and feed solutions normalized by the feed solution osmotic pressure. Data are shown for the runs at both 20 and  $30^{\circ}$ C.  $\pi_{D}$  and  $\pi_{F}$  are the osmotic pressures of the draw and feed solutions, respectively. (from Garcia-Castello *et al.*, 2009).



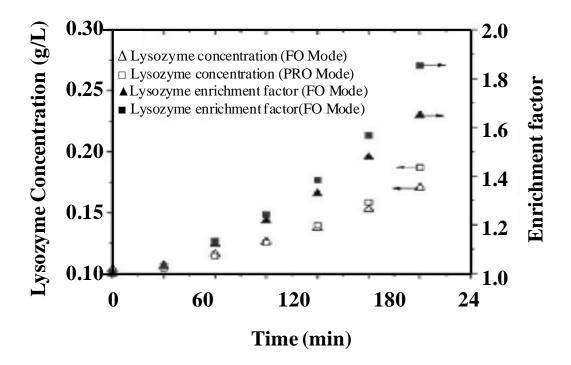
**Figure 19:** Variation of flux for (a, b) dextrose and sucrose for mode I (feed towards support layer) and mode II (feed towards active layer); (c, d) combination of sucrose, dextrose and pectin for mode I and mode II. Mode I and II refer to the feed towards support layer and feed towards active layer, respectively (from Nayak *et al.*, 2011)



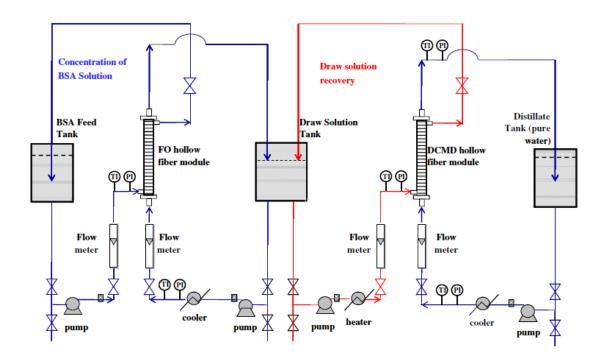
**Figure 20:** Variation of flux for real food systems (a) beetroot, (b) pineapple, (c) grape juice. (from Nayak *et al.*, 2011).



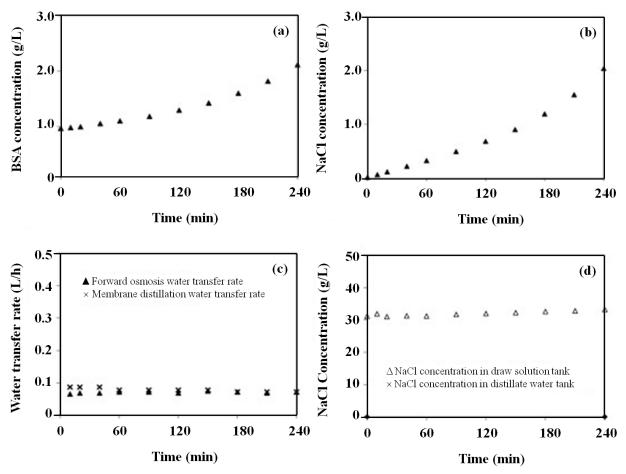
**Figure 21**: (a) Flux performance during osmotic dewatering of synthetic press liquor. (b) steady state fluxes after initial fouling for each FS solution composition. Results from the 2M NaCl and 4M NaCl draw solutions are shown as white and shaded bars, respectively. S#4 is indicative of the synthetic press liquor and acts as a reference solution containing CaCl<sub>2</sub>, pectin and citric acid along with the sugars. S#1 lacks pectin and CaCl<sub>2</sub>. S#2 lacks CaCl<sub>2</sub>. S#3 lacks pectin and S#5 lacks citric acid. In each solution, both the total organic and total inorganic content was kept constant (by mass). The pH of each solution was adjusted to 4.25 (from Garcia-Castello and McCutcheon, 2011)



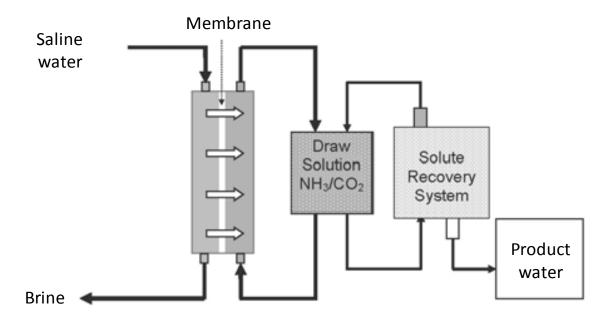
**Figure 22**: Protein enrichment using dual-layer hollow fiber forward osmosis membrane kinetics of lysozyme enrichment. (from Yang et al., 2009)



**Figure 23:** Schematic diagram of the lab-scale forward osmosis-membrane distillation hybrid system. (from Wang *et al.*, 2011).



**Figure 24**: BSA, NaCl concentrations in the feed tank, water transfer rate, NaCl concentrations in draw solution tank and distillate water tank as a function of time. Initial volume of BSA feed solution 0.6 L, BSA concentration 1.0 g/L, 28 °C, flow rate 0.025 m/s. NaCl draw solution (in FO) 0.5 M, 30 °C, flow rate 0.26 m/s. NaCl feed solution (in MD): 0.5 M, 54 °C, flow rate 0.89 m/s. Cold distillate water (in MD): 16 °C, flow rate 1.21 m/s. (from Wang et al., 2011)



**Figure 25:** Schematic diagram of the ammonia carbon dioxide forward osmosis desalination process (from McGinnes and Elimelech, 2007)

**Table 1:** Work carried out by various researchers on forward osmosis in the area of food processing

Product	Important findings	References
Grape Juice	Reverse osmosis cellulose acetate membrane was used. Sodium chloride solution was used as a draw solution. The juice was concentrated from 16 to 60° Brix with a flux of 2.5 L.m <sup>-2</sup> .h <sup>-1</sup> .	Popper et al. (1966)
	Cellulose triacetate membrane was used. 6.0 M sodium chloride was used as draw solution. The anthocyanin in grape juice was concentrated from 104.8 to 715.6 mg/L (6.8 fold) and total soluble solids from 4.4 to 54 °Brix (12.3 fold).	Nayak <i>et al</i> . (2011)
Model food system (Sucrose, dextrose and pectin)	Cellulose triacetate membrane was used. 6.0 M sodium chloride was used as draw solution. The flux for mixture of sugar and pectin in mode I and II were 4.70–2.45 L/m² h and 9.1 to 5.0 L/m² h, respectively	Nayak <i>et al.</i> (2011)
Sugar solution	Plate and frame module was used. Membrane used was made of cellulose triacetate. Sodium chloride was used as draw solution. Flux values were 5.84 and 2.78 L/m2 h for 2.0 and 4.0 M sodium chloride solution, respectively. Concentration factor obtained was 5.7°brix	Garcia-Castello <i>et al.</i> (2009)
Pineapple	Aqueous solution of sucrose (0–40%, w/w) and sodium chloride (0–26%, w/w) was used as a draw solution with a view to reduce salt transfer. Direct osmosis membranes (Osmotek Inc., Corvallis,OR) Was used The pineapple juice was concentrated up to a total soluble solids content of 60 °Brix at ambient temperature	Babu <i>et al.</i> , (2006)

The pineapple juice was concentrated in 3 cycles and the total soluble solids was found to increase from 4.4 to 54 °Brix (12 fold). Cellulose triacetate membrane was used for concentration utilizing 6.0 M sodium chloride as adraw solution.

Nayak *et al.* (2011)

Red raspberry High fructose corn syrup (HFCS) was used as draw

solution. Osmotek DOC Cell was used as a module

with an area 0.14m<sup>2</sup>. Raspberry juice was

concentrated up to 45  $^{\circ}$ Brix by forward osmosis.

Wrolstad *et al.* (1993)

Orange Raspberry Tomato Juice Poly ethylene glycol (PEG) and HFCS was used as draw solution. Module used was Osmotek DOC Cell, CTA Membrnae The maximum flux was 5-6 L/m<sup>2</sup> h.

Herron *et al.* (1994)

Tomato Juice

NaCl, CaCl<sub>2</sub>, Ca(NO<sub>3</sub>)<sub>2</sub>, PEG400, sucrose, glucose were used as draw solutions and average flux were 3.10, 2.33, 1.80, 0.70, 0.55 and 0.37 kg/m2 h, respectively. AFC99 commercial reverse osmosis membrane which was a thin film composite aromatic polyamide membrane with nominal sodium chloride rejection 99% was used.

Petrotos *et al.*, (1998)

Red Radish

The forward osmosis concentration process utilized a pilot plant unit (Osmotek Inc., Corvallis, OR). High fructose corn syrup with an initial "Brix of ca. 60" was used as an osmotic agent. A 7.5-fold concentration was achieved in forward osmosis process using two successive runs, but required 11 h. Anthocyanin

Rodriguez-Saona *et al.*, (2001)

levels of 800

mg/100 mL were obtained in concentrates with 30

"Brix.

Natural Food Colorant (Garcinia indica extract, beetroot juice) Cellulose triacetate membrane was used. The concentration of draw solution (sodium chloride solution) was varied from 1.0 - 6.0 M. The flux for mixture of sugar and pectin when feed towards support side or active layer were 4.70–2.45 L/m $^2$  h and 9.1 to 5.0 L/m $^2$  h, respectively

Nayak and Rastogi (2010a) Nayak and Rastogi (2010b)

Cellulose triacetate membrane was used. 6.0 M sodium chloride was used as draw solution. The beetroot juice was concentrated in 2 cycles and the betalains content of the juice increased from 50.92 mg/L to 2.91 g/L (57.1 fold) and total soluble solids was found to increase from 2.3 to 52 °Brix (22.6 fold).

Nayak et al., 2011

**Table 2:** Values of viscosity of the used osmotic media, the corresponding driving force and flux (from Petrotos *et al.*, 1998)

Osmotic medium	Viscosity	Δπ-ΔΡ	Flux
	in cP (25°C)	(bar)	$(kg/m^2h)$
NaCl 22.24% (w/w)	1.90	280.30	3.10
CaCl <sub>2</sub> 29.07% (w/w)	2.30	548.00	2.33
$Ca(NO_3)_2 29.00\% (w/w)$	2.90	176.30	1.80
PEG400 49.97% (w/w)	14.00	303.00	0.70
Sucrose 58.29% (w/w)	40.50	134.80	0.55
Glucose 62.86% (w/w)	103.00	337.50	0.37