A Brief Review of Reverse Osmosis Membrane Technology

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The concepts of "osmosis" and "reverse osmosis" have been known for many years. In fact, studies on osmosis were carried out as early as 1748 by the French scientist Nollet, and many researchers investigated these phenomena over the next two centuries (Reid, 1966; Mason, 1991). However, the use of reverse osmosis (RO) as a feasible separation process is a relatively young technology. In fact, only in the late 1950's did the work of Reid show that cellulose acetate RO membranes were capable of separating salt from water, even though the water fluxes obtained were too small to be practical (Reid and Breton, 1959; Ferguson, 1980; Lonsdale, 1982; Applegate, 1984). Then, in the early 1960's, Loeb and Sourirajan developed a method for making asymmetric cellulose acetate membranes with relatively high water fluxes and separations, thus making RO separations both possible and practical (Loeb and Sourirajan, 1962; Loeb, 1981; Sourirajan and Matsuura, 1985).

Since then, the development of new-generation membranes such as the thin-film, composite membrane that can tolerate wide pH ranges, higher temperatures, and harsh chemical environments and that have highly improved water flux and solute separation characteristics has resulted in many RO applications. In addition to the traditional seawater and brackish water desalination processes, RO membranes have found uses in wastewater treatment, production of ultrapure water, water softening, and food processing as well as many others (Bhattacharyya et al., 1992). An estimate indicated that sales of RO membrane products had grown to $118 million yearly in 1990, with great potential for continued growth (Riley, 1990).

The driving force for the development and use of RO membranes is the advantages that these have over traditional separation processes such as distillation, extraction, ion exchange, and adsorption. Reverse osmosis is a pressure-driven process so no energy-intensive phase changes or potentially expensive solvents or adsorbents are needed for RO separations. Reverse osmosis is a process that is inherently simple to design and operate compared to many traditional separation processes. Also, simultaneous separation and concentration of both inorganic and organic compounds is possible with the RO process. In addition, with nanofiltration ("loose RO") membranes selective solute separations based on charge and molecular weight/size differences are possible. Finally, reverse osmosis technology can also be combined with ultrafiltration, pervaporation, distillation, and other separation techniques to produce hybrid processes that result in highly efficient and selective separations (Bhattacharyya et al., 1992).

Definition of Reverse Osmosis

Osmosis is a natural phenomenon in which a solvent (usually water) passes through a semipermeable barrier from the side with lower solute concentration to the higher solute concentration side. As shown in Figure 1a, water flow continues until chemical potential equilibrium of the solvent is established. At equilibrium, the pressure difference between the two sides of the membrane is equal to the osmotic pressure of the solution. To reverse the flow of water (solvent), a pressure difference greater than the osmotic pressure difference is applied (see Figure 1b); as a result, separation of water from the solution occurs as pure water flows from the high concentration side to the low concentration side. This phenomenon is termed reverse osmosis (it has also been referred to as hyperfiltration).

A reverse osmosis membrane acts as the semipermeable barrier to flow in the RO process, allowing selective passage of a particular species (solvent, usually water) while partially or completely retaining other species (solutes). Chemical potential gradients across the membrane

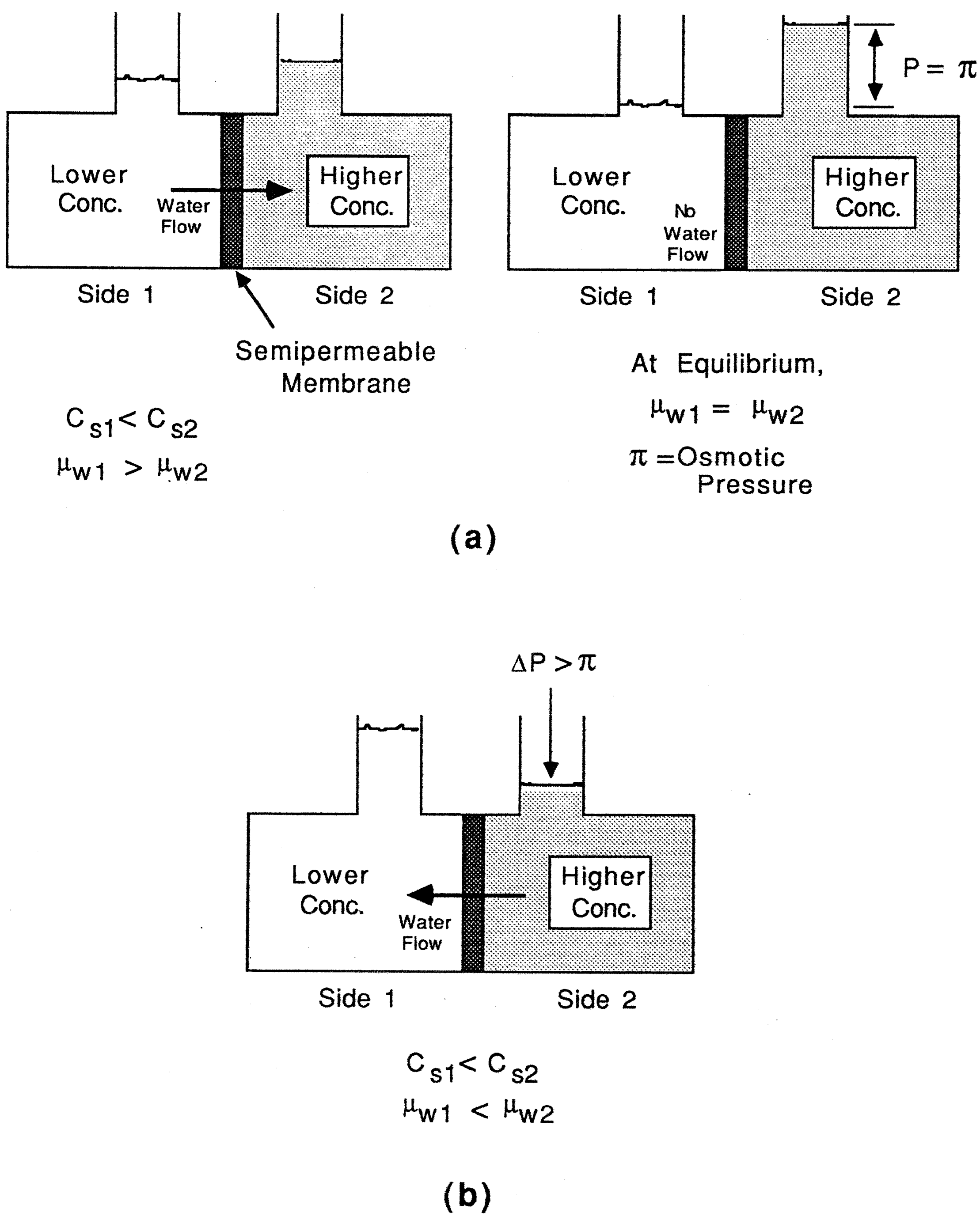

Figure 1. Schematic of Osmosis and Reverse Osmosis Phenomena.

provide the driving forces for solute and solvent transport across the membrane: $-\Delta\mu_s$, the solute chemical potential gradient, is usually expressed in terms of concentration; and $-\Delta\mu_w$, the water (solvent) chemical potential gradient, is usually expressed in terms of pressure difference across the membrane (Bhattacharyya and Williams, 1992b).

RO Process Description and Terminology

The RO process is relatively simple in design. It consists of a feed water source, feed pretreatment, high pressure pump, RO membrane modules, and, in some cases, posttreatment steps. A schematic of the RO process is shown in Figure 2a.

The three streams (and associated variables) of the RO membrane process are shown in Figure 2b: the feed; the product stream called the permeate; and the concentrated feed stream, called the concentrate or retentate. The water flow through the membrane is reported in terms of water flux, J_w, where

$$J_w = \frac{\textit{volumetric or mass permeation rate}}{\textit{membrane area}}. \qquad \text{Eqn. 1}$$

Solute passage is defined in terms of solute flux, J_s:

$$J_s = \frac{\textit{mass permeation rate}}{\textit{membrane area}}. \qquad \text{Eqn. 2}$$

Solute separation is measured in terms of rejection, R, defined as

$$R = 1 - \frac{C_P}{C_F}. \qquad \text{Eqn. 3}$$

The quantity of feed water that passes through the membrane (the permeate) is measured in terms of water recovery, r, defined for a batch RO system as

$$r = \frac{\Sigma J_w A_m \Delta t}{V_F} = \frac{V_P}{V_F}, \qquad \text{Eqn. 4}$$

and for a continuous system as

$$r = \frac{J_w A_m}{F_F} = \frac{F_P}{F_F}. \qquad \text{Eqn. 5}$$

In a batch membrane system, water is recovered from the system as the concentrate is recycled to the feed tank; as a result, if the solute is rejected the feed concentration (C_F) continuously increases over time. For a continuous membrane system, fresh feed is continuously supplied to the membrane.

Water flux is sometimes normalized relative to the initial or pure water flux (J_{wo}) as $\frac{J_w}{J_{wo}}$ or as flux drop, defined by

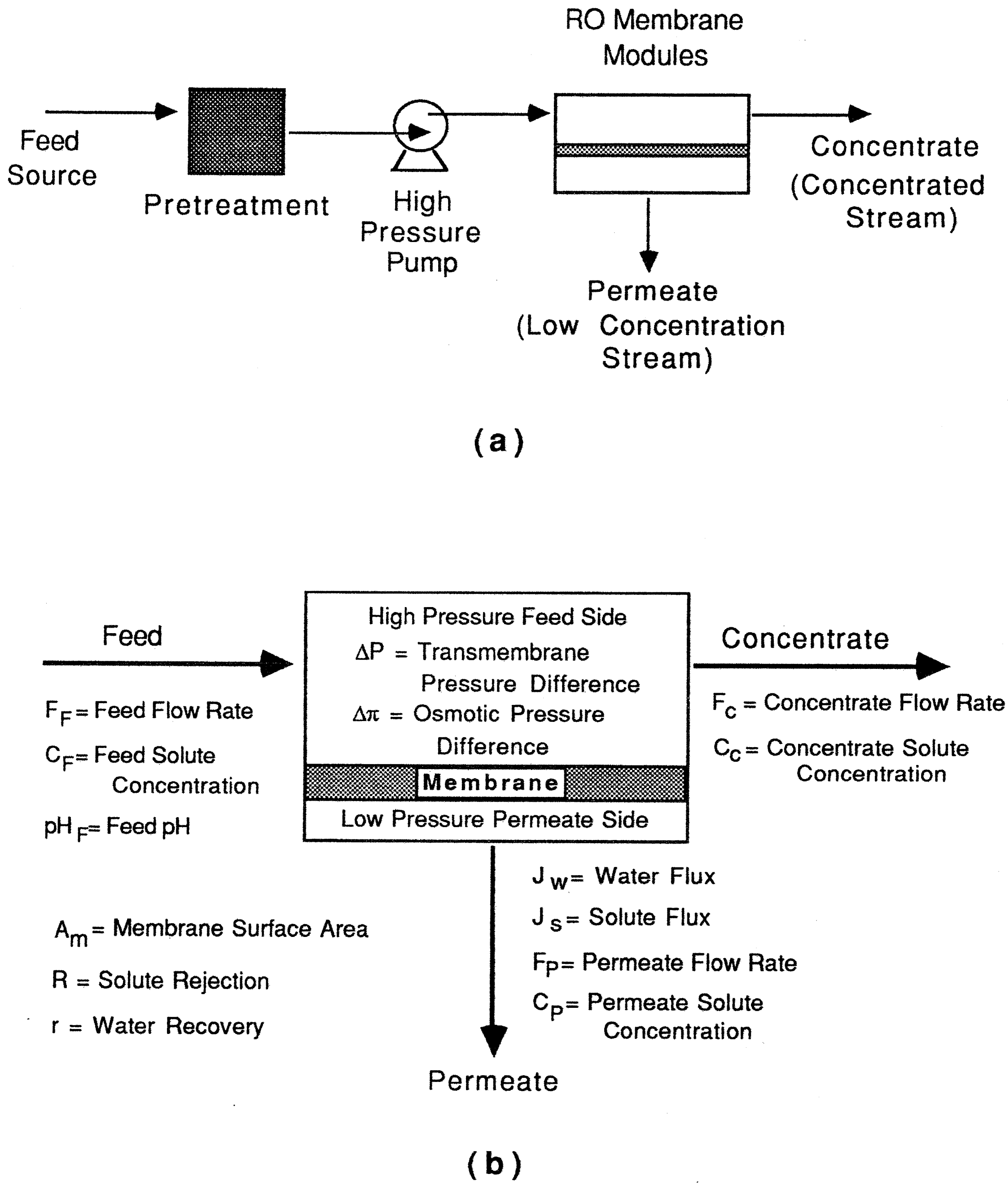

Figure 2. Schematic of (a) RO Membrane Process and (b) RO Process Streams.

$$Flux\ Drop = 1 - \frac{J_w}{J_{wo}} . \qquad \text{Eqn. 6}$$

The pressure difference between the high and low pressure sides of the membrane is denoted as ΔP while the osmotic pressure difference across the membrane is defined as $\Delta\pi$; the net driving force for water transport across the membrane is $(\Delta P - \sigma\Delta\pi)$, where σ is the Staverman reflection coefficient. Gekas (1988) has reviewed the standardized terminology recommended for use to describe pressure-driven membrane processes, including for reverse osmosis.

RO Membrane Preparation, Structures, and Properties

Reverse osmosis membrane separations are, most importantly, governed by the properties of the membrane used in the process. These properties depend on the chemical nature of the membrane material (almost always a polymer) as well as its physical structure. Properties for the ideal RO membrane include that it is resistant to chemical and microbial attack, mechanically and structurally stable over long operating periods, and have the desired separation characteristics for each particular system. However, few membranes satisfy all these criteria and so compromises must be made to select the best RO membrane available for each application. Excellent discussions of RO membrane materials, preparation methods, and structures include Cadotte et al. (1981), Kesting (1985), Lloyd and Meluch (1985), Lonsdale (1987), Cabasso (1987), Koros et al. (1988), Baker (1990), Strathmann (1990), and Petersen and Cadotte (1990).

Most currently available RO membranes fall into two categories: asymmetric membranes containing one polymer, and thin-film, composite membranes consisting of two or more polymer layers. Asymmetric RO membranes have a very thin, permselective skin layer supported on a more porous sublayer of the same polymer (see Figure 3a); the dense skin layer determines the fluxes and selectivities of these membranes while the porous sublayer serves only as a mechanical support for the skin layer and has little effect on the membrane separation properties. Since the skin layer is very thin (from 0.1 to 1 μm), the membrane resistance to water transport (which is proportional to the dense skin thickness) is much lower and, as a result, water fluxes much higher than those through comparable symmetric membranes (Lonsdale, 1987; Baker, 1990; Strathmann, 1990).

Asymmetric membranes are most commonly formed by a phase inversion (polymer precipitation) process. In this process, a polymer solution is precipitated into a polymer-rich solid phase that forms the membrane and a polymer-poor liquid phase that forms the membrane pores or void spaces. The rate of precipitation is a factor in determining pore characteristics: a rapid precipitation tends to produce pores that are small and asymmetric while slow precipitation produces more symmetrical pores that are relatively large (Kesting, 1985; Cabasso, 1987; Baker, 1990; Strathmann, 1990). The polymer precipitation can be achieved in several ways, including thermal gelation, solvent evaporation, or nonsolvent immersion, but nonsolvent immersion is the most important asymmetric membrane preparation technique; this is the Loeb-Sourirajan preparation method that was employed to form the first asymmetric cellulose acetate membranes. In this technique a polymer solution is cast into a film and then the polymer precipitated by immersion into a nonsolvent; the nonsolvent (water, for example) rapidly precipitates the polymer on the surface of the cast film, forming the very thin, dense skin layer of the membrane. The polymer beneath the skin layer precipitates more slowly, resulting in a more porous polymer sublayer (Kesting, 1985; Cabasso, 1987; Baker, 1990; Strathmann, 1990). Following polymer precipitation, the membrane is

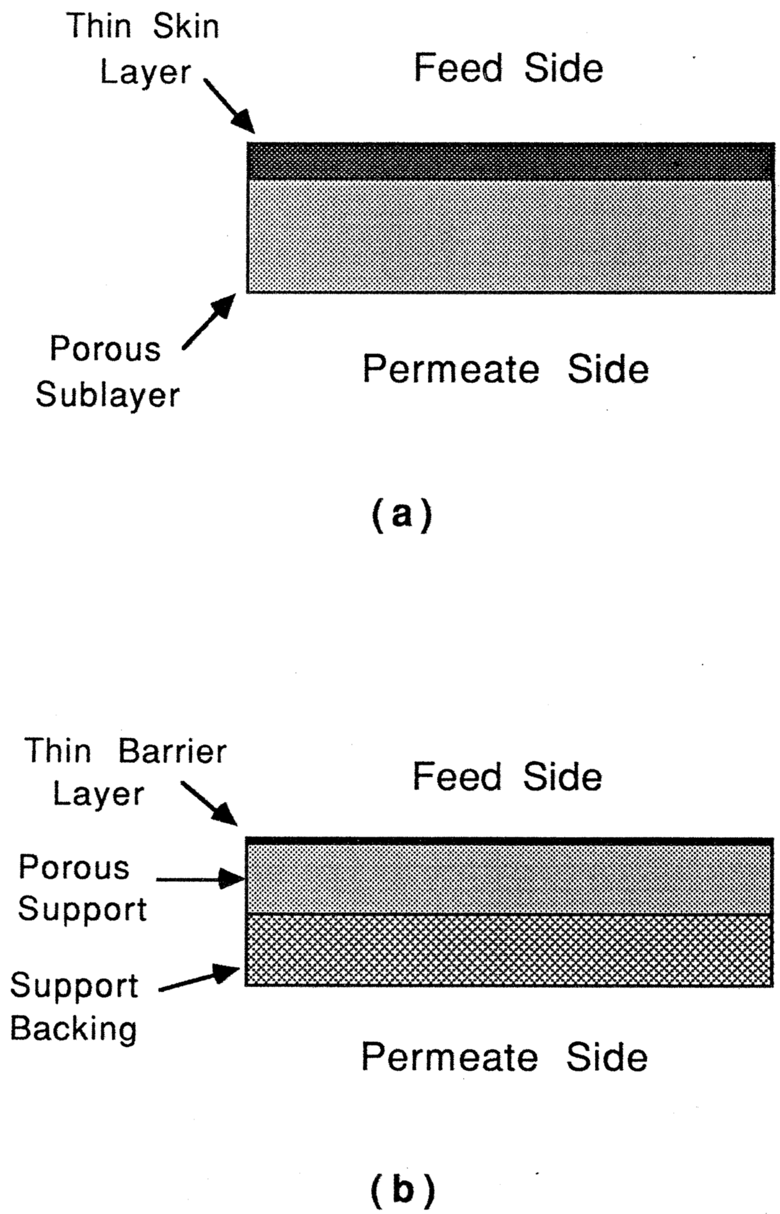

Figure 3. Schematic of (a) Asymmetric Membrane and (b) Thin-Film Composite Membrane.

usually annealed in order to improve solute rejection. Widely used examples of asymmetric membranes include cellulose acetate membranes and linear aromatic polyamide membranes.

Thin-film, composite membranes consist of a thin polymer barrier layer formed on one or more porous support layers (almost always a different polymer from the surface layer). Figure 3b shows a schematic of this type of membrane. The surface layer determines the flux and separation characteristics of the membrane; the porous backing serves only as a support for the barrier layer and so has almost no effect on membrane transport properties. The barrier layer is extremely thin, on the order of 0.1 μm or less, thus allowing high water fluxes (Cadotte et al., 1981; Lonsdale, 1987; Baker, 1990; Strathmann, 1990; Petersen and Cadotte, 1990).

The most important thin-film, composite membranes are made by interfacial polymerization, a process in which a highly porous membrane (usually polysulfone) is coated with a polymer or monomer and then reacted with a cross-linking agent. A dense, cross-linked polymer layer forms at the solution interface, and, since the cross-linking reaction occurs mostly at the solution interface, the resulting barrier layer is extremely thin. A less cross-linked, more permeable layer forms under the surface layer and fills the pores of the support membrane (Cabasso, 1987; Baker, 1990; Strathmann, 1990; Petersen and Cadotte, 1990). These thin, highly cross-linked polymer membranes can have much higher selectivities and water fluxes compared to the asymmetric type since the barrier layers of the composite membranes are usually much thinner than those of the asymmetric membranes. One of the most widely-used thin-film, composite membranes consists of cross-linked aromatic polyamide on a polysulfone support layer.

The exact nature of the structure of the thin skin of asymmetric or thin-film, composite RO membranes is unclear and is still a point of debate. In order to model RO membrane separations, some researchers have viewed the skin as a homogeneous film of polymer containing no pores or voids unless these are present as imperfections (Lonsdale et al., 1965; Sherwood et al., 1967; Burghoff et al., 1980; Pusch, 1986; Soltanieh and Gill, 1981; Bitter, 1991). They assume that solvent or solute transport occurs through the interstitial spaces of the polymer chains. Other researchers have assumed that the barrier layer is microporous; that is, extremely small pores or voids (usually <30 A radius) are formed during casting and transport occurs through these pores (Merten, 1966; Jonnson and Boesen, 1975; Soltanieh and Gill, 1981; Sourirajan and Matsuura, 1985; Bhattacharyya et al., 1986; Mehdizadeh and Dickson, 1989). However, others have considered a more complex view of the barrier layer in RO membranes. Kesting (1990) postulated that the layer consists of polymer nodules (clusters of polymer macromolecules) and nodule aggregrates; he indicated that transport can occur through chain segment displacements in the polymer nodules (interstitial spaces) and through spaces between nodule aggregates (defect pores). In addition, Tam et al. (1991) considered the fractal (random) nature of pore distribution and geometry in the barrier layer; their analysis recognized the randomness that could occur during formation of the barrier layer pores. Even though the simpler concepts (homogeneous versus pores) describing the barrier have been used successfully in model development, the approaches of Kesting and Tam et al. probably represent more realistic descriptions of the barrier layer. However, as of yet, no technique is available to exactly determine the barrier layer structure.

Although RO membranes have been formed and tested with a wide range of different materials and preparation techniques, the cellulosic polymers (cellulose acetate, cellulose triacetate, etc.), linear and cross-linked aromatic polyamide, and aryl-alkyl polyetherurea are among the most important RO membrane materials (Pusch and Walch, 1990; Riley, 1990; Strathmann, 1990; Bhattacharyya et al., 1992). Asymmetric cellulose acetate membranes continue to enjoy widespread use despite some disadvantages: a narrow pH operating range (4.5-7.5) since it is subject to hydrolysis; susceptibility to biological attack; compaction (mechanical compression) at high pressures which results in reduced water flux; and low upper temperature limits (~35 °C). Polyamide and polyurea composite membranes typically have higher water fluxes and salt and organic rejections, can withstand higher temperature and larger pH variations (4-11), and are immune to biological attack and compaction. However, these membranes tend to be less chlorine-resistant and more susceptible to oxidation compared to cellulose acetate membranes; these can also be more expensive (Cadotte et al, 1981; Applegate, 1984; Riley, 1990; Sudak, 1990; Bhattacharyya et al., 1992).

Nanofiltration (or "loose RO") membranes are a relatively recent development in the field of RO membrane separations. These membranes typically have much higher water fluxes at low pressures compared to traditional RO membranes. Nanofiltration membranes are usually charged (carboxylic groups, sulfonic groups, etc.), and, as a result, ion repulsion (Donnan exclusion) is the major factor in determining salt rejection; that is, more highly charged ions such as SO_4^{2-} are more highly rejected than monovalent ions such as Cl^- by a negatively-charged nanofiltration membrane. These membranes also usually have good rejections of organic compounds with molecular weights above 200 to 500 (Eriksson, 1988; Cadotte et al., 1988; Williams et al., 1992). The most important nanofiltration membranes are composite membranes made by interfacial polymerization; aromatic polypiperazine is an example of a widely-used nanofiltration membrane.

There are many commercially available RO membranes, both of the asymmetric and thin-film, composite type, and these membranes have a wide variety of water flux and rejection properties. Figure 4 shows water flux and NaCl rejections for three different classes of RO membranes (nanofiltration, low pressure RO, and high pressure RO) made from a variety of polymer materials. Bhattacharyya et al. (1992) also list selected solute (both inorganic and organic) rejections for a large number of RO membranes.

RO Membrane Modules and Module Configurations

While the membrane material largely determines the water and solute fluxes in a RO process, Bhattacharyya et al. (1992) pointed out that the packaging of the RO membrane is also extremely important to the feasibility of the process. The requirements of a membrane module include (Bhattacharyya et al., 1992): (1) that it offer mechanical support to the fragile RO membrane even at high operating pressures (up to 8 MPa); (2) that the design minimize pressure drop across the module as well as fouling and concentration polarization; and (3) that the module be relatively inexpensive and easy to replace in the membrane process. The most common commercially available membrane modules include plate-and-frame, tubular, spiral-wound, and hollow-fiber elements.

Plate-and-frame modules consist of stacks of flat sheet membrane placed on supports; each membrane and support are separated by spacers which direct the feed across each membrane and channel the permeate out of the module (Allegrezza, 1988; Baker, 1990; Strathmann, 1990; Bhattacharyya et al., 1992). While this module is resistant to fouling, it has a low membrane surface area per element (defined as packing density); this makes it expensive and can limit its use in areas with space restrictions. Tubular membrane elements consist of membrane tubes (typically 1.3 cm in diameter) supported within perforated stainless steel tubes; as feed flows through the tubes, the permeate passes through the membrane and support (Allegrezza, 1988; Bhattacharyya et al., 1992). While these elements are also fouling resistant and are easy to clean, the modules have a low packing density and can be expensive to operate because of the high feed flow rates necessary. Because of the plate-and-frame and tubular element disadvantages, these modules are used

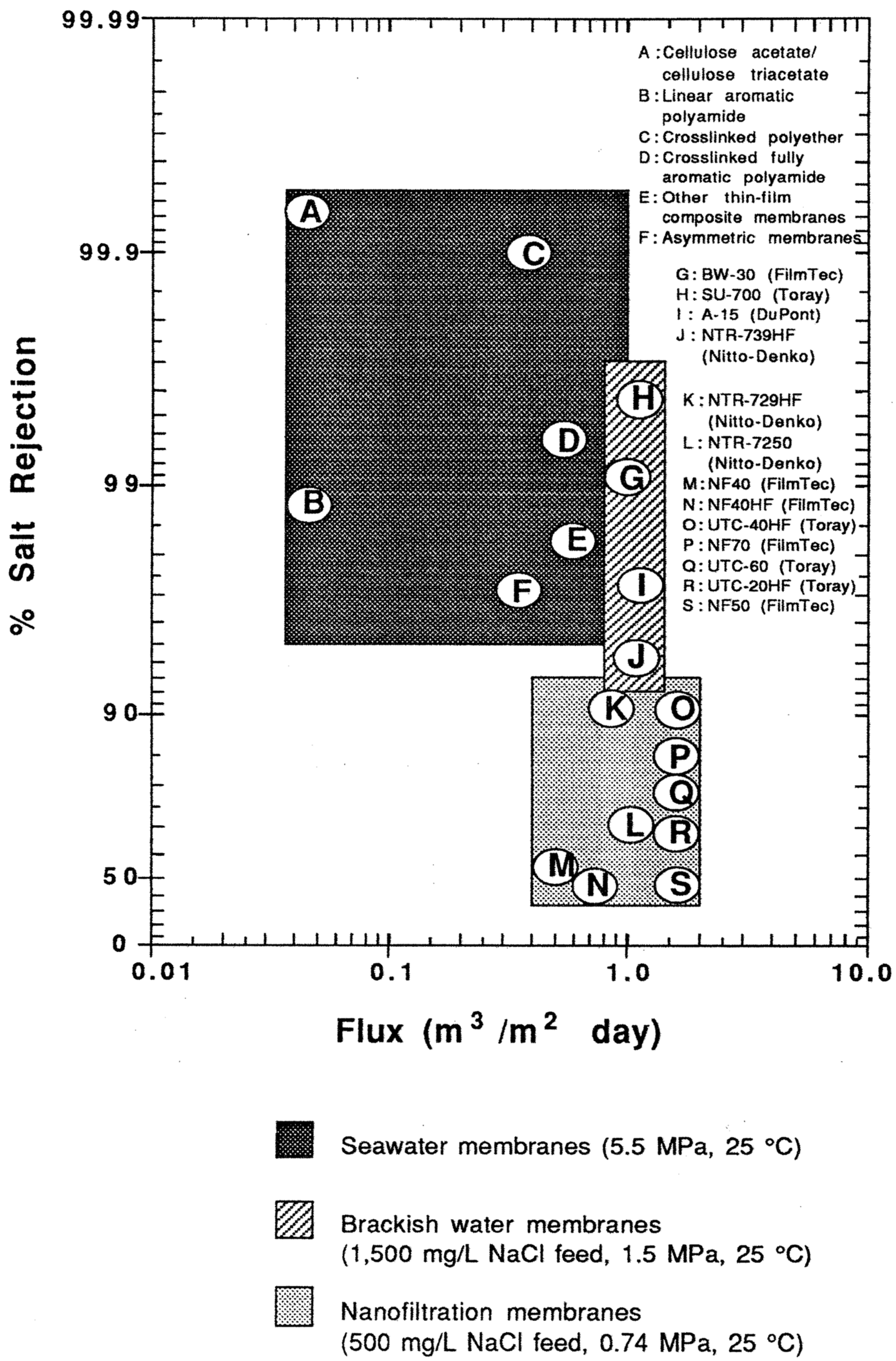

Figure 4. Water Flux and NaCl Rejections for Nanofiltration, Low Pressure RO, and High Pressure RO Membranes (Adapted from Riley, 1990[1]).

[1] Intended to show relative performances of classes of membranes only. Actual performance may vary widely depending upon feed conditions, etc. In addition, newer classes/types of membranes may out perform those shown.

primarily for highly fouling feeds.

The most widely used membrane modules are the spiral-wound and hollow-fiber elements. A spiral-wound element consists of flat sheets of membrane separated by spacers that are rolled around a perforated collection tube. The feed is channeled across these rolled membrane sheets, permeates through the membrane, and is collected in the center tube (Allegrezza, 1988; Bhattacharyya et al., 1992). This type of element has a high packing density, moderate fouling resistance, and lower capital and operating costs compared to plate-and-frame or tubular modules. Hollow-fiber elements consists of large numbers of fine hollow fiber membranes (with an outer diameter up to 200 μm) placed in a pressure vessel; the feed flows outside the fibers and permeates through these (Allegrezza, 1988; Baker, 1990; Bhattacharyya et al., 1992). These elements have an extremely high packing density and so can have high permeate production rates per module. However, these modules are highly prone to fouling and so are not feasible for some applications.

Because of the importance of the membrane module used in the RO process, much research has been performed to optimize the design of each element. As a result, many models describing the various modules are available, allowing determination of module hydrodynamics, optimal membrane spacer placement, hollow-fiber diameter, etc. Excellent discussions of membrane module design and modelling methods include Doshi (1988), Rautenbach and Albrecht (1989); and Bhattacharyya et al. (1992).

Reverse osmosis membrane modules can be arranged in several configurations in the RO process (Williams et al., 1992). For a single-pass arrangement, a single high rejection membrane sufficiently removes the solute from the feed. In a double-pass configuration, the permeate of one set of membranes is used as the feed to another set of membranes in order to provide adequate overall removal of the solute. The modules can also be placed in stages in order to increase water recoveries; in this configuration, the concentrates from one set of membranes is used as the feed for another set and so high overall water recoveries are possible.

RO Membrane Characterization Techniques

Characterization of RO membranes is important since this allows insight into the relationship between membrane chemistry, structure, and transport properties. The most widely used characterization method is the measurement of water flux and solute (usually NaCl) rejection for the membrane; these can be easily measured and so give a quick indication of the suitability of a particular membrane for an application. However, fluxes provide only limited information about the characteristics and structure of the membrane and the role these play in water and solute transport. As a result, other characterization techniques are beginning to be employed in order to determine parameters such as pore size, barrier layer thickness, and membrane elemental composition.

Simon and Calmon (1986) and Pusch (1986) discuss the measurement of several RO membrane characteristics, including overall membrane thickness, water content, membrane potential, ionic exchange capacity, etc. In addition, Jevtitch (1986), Bhattacharyya et al. (1986), Han (1989), and Han and Bhattacharyya (1991) described the use of vapor adsorption data of carbon dioxide and nitrogen gases in order to determine pore volumes and pore size distributions for cellulose acetate and composite aromatic polyamide membranes. Alternatively, several researchers have used experimental flux data and solute-membrane interaction parameters in order to calculate pore sizes and distributions (Jonnson and Boesen, 1975; Sourirajan and Matsuura, 1985; Mehdizadeh and Dickson, 1989). Glaves and Smith (1989) indicated that nuclear magnetic resonance (NMR) may also be suitable for determining membrane pore structures. Kesting (1985), Cabasso (1987), Strathmann (1990), Petersen and Cadotte (1990), and Kesting (1990) have described scanning electron micrographs (SEM) for asymmetric and composite membranes. Although they indicated no information on the barrier layer pore structure was discernible from the micrographs, they pointed out that the asymmetric or composite nature of the membranes was clearly visible and that it was possible to approximate the barrier layer thickness from the micrographs. Bartels (1989) also examined the membrane barrier layer for composite membranes with both SEM and transmission electron microscopy (TEM).

Considerable attention has been given to the application of spectroscopic techniques to the characterization of RO membranes. Bartels (1989) examined RO membranes using infrared (IR) spectroscopy; he found that IR provided valuable information on the functional groups (such as carboxylic acid or amide groups) present in the composite membrane studied. Arthur (1989) made similar studies with several different composite RO membranes, and Avlonitis et al. (1992) studied changes in aromatic polyamide membranes caused by chlorine degradation by following changes in the membranes IR spectra. Koo et al. (1986), Bartels (1989), and Arthur (1989) used X-ray photoelectron spectroscopy (XPS), sometimes referred to as ESCA, to study elemental compositions of composite RO membranes near the surface; this technique supplied verification of the polymer chemical structures expected from the interfacial polymerization reactions that formed the membranes. Bartels (1989) also used Rutherford backscattering spectroscopy (RBS) to determine elemental composition; results were similar to those obtained by XPS.

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