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INTRODUCTION

Scope

The separation, concentration, and purification of molecular mixtures are major unit operations in the chemical industries. Efficient separation processes are needed to achieve high-grade products in the food and pharmaceutical industries, to suuply communitites and industry with high-quality water, and to remove or recover toxic or valuable components from industrial effluents. For this task a multitude of separation techniques such as distillation, precipitation, crystallization, extraction, adsorption, and ion exchange are used today. More recently, these conventional separation methods have been supplemented by a family of processes that utilize semipermeable membranes as separation barriers.

Membranes and membrane processes were first introduced as an analytical tool in chemical and biochemical laboratories, and they developed very rapidly into industrial products and methods with significant technical and commercial impact. Today, membranes are used on a large scale to produce potable water from seawater, to clean industrial effluents and recover valuable constitutents, to concentrate, purify, or fractionate macromolecular mixtures in the food and drug industries, and to separate gases and vapors. They are also key components in energy conversion and storage systems, artificial organs, and drug delivery service. (13)

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General Design Considerations

The membranes used in the various applications differ widely in their structure, their function, and the way they are operated in a separation process. However, all membrane processes share several features that make them particularly attractive tools for the separation of molecular mixtures. The separation is performed by physical means at ambient temperature without chemically altering the constituents. This is important for applications in artificial organs and in many drug delivery systems, as well as in the food and drug industries or in downstream processing of bioproducts where specific separation tasks, and membrane processes are often technically simpler and more energy efficient than conventional separation techniques and are eqully well suited for large-scale continous operations as for batchwise treatment of very small quantities.

Key property of a membrane is permselectivity, which is determined by differences in the transport rate of various components through the membrane. The transport rate of a component through a membrane is determined by the structure of the membrane, the size of the permeating component, by the chemical nature and the electrical charge of the membrane material and permating component, by the driving force due to the chemical or eletrochemical potential gradients, that is, concentration pressure and electrical potential differences.

Some driving forces such as pressure, temperature, and concentration gradients act equally on all components. The use of different membrane structures and driving forces has resulted in a number of rather different membrane processes such as reverse osmosis, micro- and ultrafiltration, dialysis, electrodialysis, donnan dialysis, pervaporation, and gas separation. The versality of membrane structures and functions makes a precise and complete definition of a membrane difficult.

Generally, a membrane can be defined as a barrier that separates two different regions and controls the exchange of matter, energy, and information between the regions in a very specific way. The term "membrane" described in two very different items, first one is biological membranes, which are part of a living organism, and the second is synthetic membranes. Synthetic membranes are not nearly as complex in their structure or function as biological membranes. They are usually less selective and energy-efficient but have significantly higher chemical, thermal, and mechanical stability.

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Today membranes are used on a large technical scale in three distinct areas: First, including applications in which the use of membranes is technically feasible, but where they must compete with conventional separation processes on the basis of overall economy. This, for instance, is the case in seawater desalination and the treatment of certain wastewater streams. Here, membranes must compete with distillation and biological treatment, respectively.

The second area includes applications for which alternative techniques are available, but membranes offer a clear technical and commercial advantage. This is the case in the production of utlrapure water and in the separation of certain food products. Finally, there are applications where there is no reasonable alternative to membrane processes. This is the case in the certain drug delivery systems and artificial organs.

Since the 1960s, membrane science has grown from a laboratory curiosity to a widely practiced technology in industry and medicine. This growth is likely to continue for sometime, particularly in the membrane gas separation and pervaporation separation areas. Membranes will play a critical role in the next generation of biomedical devices, such as the artificial pancreas and liver. The total membrane market grew from \$10 million to the \$1-2 billion level in the 30 years prior to 1994. Spectacular growth of this magnitude is unlikely to continue, but a doubling in the size of the total industry to the \$2-4 billion level during the decade following is likely. Some industry use membrane system for their process. Membrane types will be discussed detail in theory section.

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Table 1: Applications of membrane separation system

Type of membrane	Applications
Reverse Osmosis	 Desalinization of brackish water Treatment of wastewater to remove a wide variety of impurities Treatment of surface and ground water Concentration of foodstuffs Removal of alcohol from beer and wine
Dialysis	 Separation of nickel sulfate from sulfuric acid Hemodialysis (removal of waste metabolites, excess body water, and restoration of electrolyte balance in blood)
Electrodialysis	 Production of table salt from seawater Concentration of brines from reverse osmosis Treatment of wastewaters from electroplating Demineralization of cheese whey Production of ultrapure water for semiconductor industry
Microfiltration	 Sterilization of drugs Clarification and biological stabilization of beverages Purification of antibiotics Separation of mammalian cells from a liquid
Ultra-filtration	 Pre-concentration of milk before making chees Clarification of fruit juice Recovery of vaccines and antibiotics from fermentation broth Color removal from kraft black liquor in paper making
Prevaporation	 Dehydration of ethanol-water azeotrop Removal of water from organic solvents Removal of organics from water

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Gas permeation	 Separation of CO₂ or H₂ from methane and other hydrocarbons Adjustment of the H2/CO ration in synthesis gas Separation of air into nitrogen and oxygen enriched streams Recovery of helium Recovery of methane from biogas
Liquid membrane	 Recovery of zinc from wastewater in the viscose fiber industry Recovery of nickel from electroplating solutions

Membrane separations represent a new type of unit operation. The membrane acts as a semipermeable barrier and separation occurs by the membrane controlling the rate of movement of various molecules between two liquid phases, two gas phases, or a liquid and a gas phase. The two fluid phases are usually miscible and the membrane barrier prevents actual, ordinary hydrodynamic flow.

Classification of membrane processes

1. Porous membrane

- a. Gas diffusion. The rates of gas diffusion depend on the pore sizes and the molecular weights.
- b. Microfiltration (MF). This refers to membranes that have pore diameters from 0.1 to 10 m. It is used to filter suspended particulates, bacteria or large colloids from solution.
- c. Ultrafiltration (UF). This refers to membranes having pore diameters in the range 20-1000 Å . It can be used to filter dissolved macromolecules, such as proteins and polymers, from solution.
- d. Reverse osmosis (RO). The membrane pores are in the range of 5-20 Å in diameter, which are within the range of the thermal motion of the polymer chains.
- e. Dialysis

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- 2. Tight (nonporous, or dense) membrane Here the permeants are sorbed into the membrane material under the influence of their thermodynamic potential and pass it as a result of a driving force exerted:
 - a. Gradient of vapor pressure pervaporation (feed is liquid) vapor permeation (feed is vapor)
 - b. Pressure gradient gas permeation (feed & permeant are gases) reverse osmosis (feed & permeant are liquids)
 - c. Temperature gradient thermoosmosis
 - d. Concentration gradient dialysis (osmosis, liquid permeation) pertraction
 - e. Gradient in electric potential electrodialysis (ion-selective membrane)

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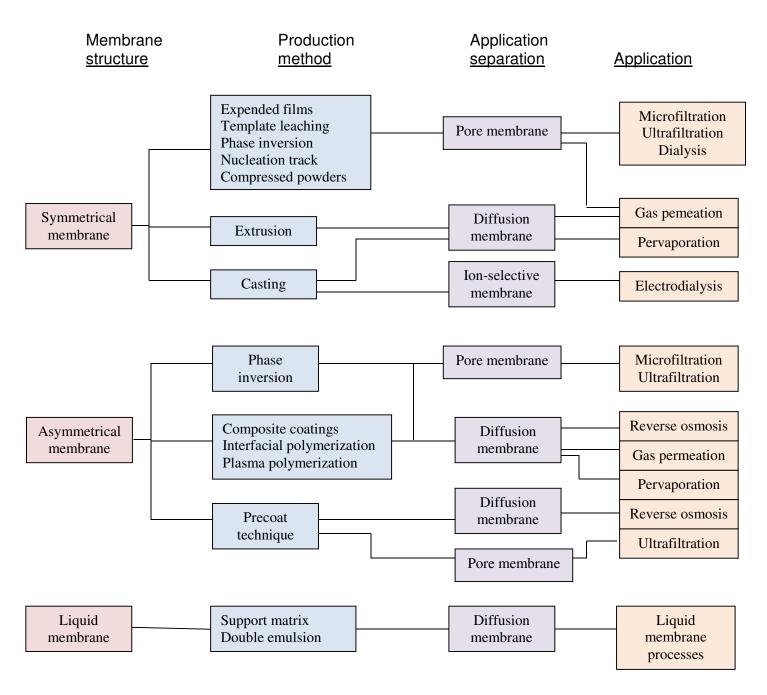


Figure 1: Membrane classification

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Membrane Materials

Almost all industrial membrane processes are made from natural (wool, rubber, cellulose) or synthetic polymers. At low temperatures typically below 100C, idealized polymers can be classified as glassy or crystalline. Glassy type is brittle and glassy in appearance and lacks any crystalline structure while the crystalline type is brittle, hard, and stiff with a crystalline structure. If the temperature of a glassy polymer is increased the glass transition temperature (T_g) may be reached where the polymer becomes rubbery. If the temperature of a crystalline polymer is increased, the melting temperature (T_m) is reached where the polymer becomes melted.

Most polymers have both amorphous and crystalline regions that is a certain degree of crystallinity that varies from 5-90%, making it possible for some polymers to have both a $T_{\rm g}$ and $T_{\rm m}$. Membranes made of glassy polymers can operate below or above $T_{\rm g}$ while membrane of crystalline polymers must operate below $T_{\rm m}$.

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Table 2: Common polymers used in membranes

Polymer	Туре	Glass Transition Temp. °C	Melting Temp.	Characteristic
Cellulose triacetate	Crystalline	Tempt 0	300	• Consist of a thin dense skin 0.1 – 1.0 µm (permslective layer)
Polyisoprene/natural rubber	Rubbery	-70		
Aromatic polyamide	Crystalline		275	 Long-term thermal stability High resistance to solvent Some are easily fabricated into fibers, films, and sheets
Polycarbonate	Glassy	150		ThermoplasticCan be extruded into various shapes
Polyimide	Glassy	310 – 365		 Tough, amorphous polymer High resistance to heat and excellent wear resistance
Polystyrene	Glassy	74 – 110		 Linear, amorphous, highly pure polymer 1000 unit Can be annealed (heated and then cooled slowly) convert to a crystalline polymer
Polysulfone	Glassy	190		Easily spun into hollow fibersHigh strength
Polytetrafluoroethylene	Crystalline		327	 Highly crystalline polymer possess exceptional thermal stability Can be formed into sheet, films, and tubing

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Types of Membrane

A membrane is a discrete, thin interface that moderates the permeation of chemical species in contact with it. This is interface may be molecularly homogenous, that is, completely uniform in composition and structure, or it may be chemically or physically heterogenous, for example, containing holes or pores of finite dimensions. A normal filter meets this definition of a membrane, but, by convention, the term membrane is usually limited to structures that permeates dissolved or collodial species, whereas the term filter is used to designate structures that separate particulate suspensions. The principal types of membrane are shown schematically in Figure 1.

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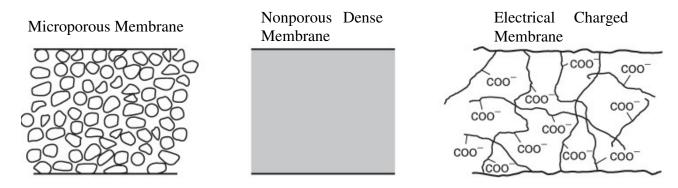
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Symmetrical Membrane



Anisotropic Membrane

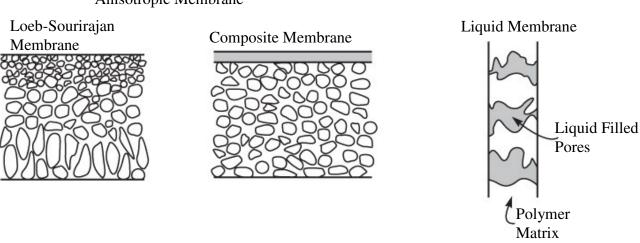


Figure 2: Principal types of membrane (Baker, 2004)

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Synthetic membranes show a large variety in their physical structure and the materias they are made from. Based on their structure they can be classified into four groups :

- Porous membranes.
- Homogenous solid membranes.
- Solid membranes carrying eletrical charges.
- Liquid or solid films containing selective carriers.

Furthermore, the structure of membranes may be symmetric, that is, the sturcture is identical over the entire cross section of the membrane, or it may be asymmetric, that is, the structure varies over the cross section of the membrane. The materials used for the preparation of membranes can be polymers, ceramics, glass, metals, or liquids. The materials may be neutral or carry electrical charges (for example, fixed ions).

1. Isotropic Microporous Membranes.

A porous structure represents a very simple form of a membrane, which closely resembles the convetional fiber filter as far as the mode of separation is concerned. Separation of the various components is achieved strictly by a sieving mechanism with the pore diamteres and the particle sizes being the determining parameters.

Porous membranes can be made from various materials like ceramics, graphite, metals, metal oxides, and various polymers. The structure can be symmetric, that is, the pore diameters do not vary over the membrane section, or asymmetric, that is, the pore diameters increase drom one side of the membrane to the other, typically by a factor of 10 to 1000. The techniques for the preparation of porous membranes can be rather different and include simple pressing and sintering of polymer or ceramic powders, irradiation and leaching of templates, as well as phase-inversion and polymer precipitation proceudres or sol – gel conversion techniques.

A microporous membrane is very similar in its structure and function to a conventional filter. It has a rigid, highly voided structure with randomly distributed, interconnected pores. However, these pores differ from those in a conventional filter by being extremely small, of the order of $0.01-10~\mu m$ in diameter. All particles smaller than the largest pores, but larger than the smallest pores are partially rejected, according to the pore size distribution of the membrane. Particles much smaller than the smallest pores pass through the membrane. Thus separation of solutes by microporous membranes is mainly a function of molecular

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size and pore size distribution. Generally, only molecules that differ considerably in size can be separated effectively by microporous membranes like in ultrafiltration and microfiltration.

2. Nonporous Dense Membranes.

Nonporous, dense membranes consist of a dense film through which permeants are transported by diffusion under the driving force of a pressure, concentration, or electrical potential gradient. The separation of various components of a solution is related directly to their relative transport rate within the membrane, which is determiend by their diffusity and solubility in the membrane material. An important property of nonporous, dense membranes is that even permeants of similar size may be separated when their concentration in the membrane material (for example, their solubility) differ significantly. Most gas separation, pervaporation, and reverse osmosis membranes use dense membranes to perform the separation. However, these membranes usually have an asymmetirc structure to improve the flux.

3. Electrically Charged Membranes.

Eletrically charged membranes can be dense or microporous, but are most commonly microporous, with the pore walls carrying fixed positively or negatively charged ions. A membrane with positively charged ions is referred to as an anion-exchange membrane because it binds anions in the surrounding fluid. Similarly, a membrane containing negatively charged ions is called a cation-exchange membrane. In a cation-exchange membrane, the fixed anions are in electrical equilibrium with mobile cations in the interstices of the polymer. On the contrary, the mobile anions are more or less completely excluded from the cation-exchange membrane because of their electrical charge, which is identical to that of the fixed ions.

Separation with charged membranes is achieved mainly by exclusion of ions of the same charge as the fixed ions of the membrane structure, and to a much lesser extent by the pore size. Due to the exclusion of the anions, a cation-exchange membrane permits transfer of cations only. Anion exchanger membranes carry positive charges fixed on the polymer matrix. Therefore, they exclude all cations and are permeable only to anions.

Films carrying charged groups are referred to as ion-exchange membranes. They consist of highly swollen gels carrying fixed positive or negative charges. The properties and

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preparation procedures of ion-exchange membrane are closely related to those of ion-exchange resins. The separation is affected by the charge and concentration of the ions in solution. For an example, monovalent ions are excluded less effectively that divalent ions and, in solutions of high ionic strength, selectivity decreases.

Although there are a number of inorganic ion-exchange materials, mostly based on zolites and bentonites, these materials are rather unimportant in ion-exchange membranes compare to polymer materials. Electically charged membranes are used for processing electrolyte solutions in electrodialysis. They aare also used as ion-conducing separators in batteries and fuel cells.

4. Asymmetric Membranes.

The transport rate of a species through a membrane is inversely proportional to the membrane thickness. High transport rates are desirable in membrane separation processes for economic reasons, therefore, the membrane should be as thin as possible. Conventional film fabrication tehcnology limits manufacture of mechanically strong, defetfree films to about 20 μ m thickness. The develoment of novel membrane fabrication techniques to produce asymmetric membrane structures was one of the breakthroughs of membrane technology during the past 30 years.

Asymmetric membranes consist of an extremely thin surface layer supported on a much thicker porous, dense substructure. The surface layer and its substructure may be formed in a single operation or formed separately. The separation properties and permeation rates of the membrane are determined exclusively by the surface layer, the substructure functions as a mechanical support. The advantages of the higher fluxes provided by asymmetric membranes are so great that almost all commercial processes use such membranes.

In a symmetric membrane the structure and the transport properties are identical over the entire cross section, and the thickness of the entire membrane determines the flux. In asymmetric membranes structural and transport properties vary over the membrane cros section. An asymmetric membrane consists of a $0.1-1~\mu m$ thick 'skin' layer on a highly porous $100-200~\mu m$ thick substructure. The skin represents the actual selective barrier of the asymmetric membrane. Its separation characteristics are determined by the nature of the material or the size of its pores. The mass flux is determined mainly by the skin thickness. The porous sublayer serves only as a support for the mosly thin and fragile skin

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and has little effect on the separation characteristics or the mass transfer rate of the membrane.

Asymmetric membranes are used primarily in pressure-driven membrane processes such as reverse osmosis, ultrafiltration, and gas and vapor separation, since here the unique properties of asymmetric membranes, that is, high fluxes and good mechanical stability, can be utilized. Two techniques are used to prepare asymmetric membranes: One utilizes the phase-inversion process, which leads to an intergral structre with the skin and the support structure made from the same material in a single process, and the other resembles a composite structure where a thin barrier layer is deposited on a porous substructure in a two-step process. In this case barrier and uspport structures are generally made from different materials.

5. Ceramic, Liquid, and Metal Membranes

This part implies that membranes material are organic polymers and, in fact, the vast majority of membranes used commercilay are polymer based. However, interest in membranes formed from less conventional materials has increased. Ceramic membranes, a special class of microporous membranes, are being used in ultrafiltration and microfiltration applications, for which solvent resistance and thermal stability are required. Dense metal membranes, particularly palladium membranes, are being considered for the separation of hydrogen from gas mixtures, and supported emulsified liquid films are being developed for coupled and facilitated transport processes.

Liquid membranes are mainly used in combination with the so-called facilitated transport which is based on "carriers" which transport certain components such as metal ions selectivity across the liquid membrane interphase. Generally, it is no problem to form a thin fluid film. It is difficult, however, to maintain and control this film and its properties during a mass separation process. To avoid breakup of the film, some type of the reinforcement is necessary to support such a weak membrane structure.

Two different techniques are used today for the prepartion of liquid membranes. First, the selective liquid barrier material is stabilized as a thin film by a surfactant in an emulsion-type mixture. In the second, a porous structure is filled with the liquid membrane phase. Both types of membranes are used today on a pilot-plant scale for the selective removal of heavy metal ions or certain organic solvents from industrial waste streams. They have also been used rather effectively for the separation of oxygen and nitrogen.

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Advantages and Limitations

In many applications membrane processes compete directly with the more conventional techniques. However, compared to these conventional procedures, membrane processes are often more energy efficient, simpler to operate, and yield higher quality products. Furthermore, the environmental impact of all membrane processes is relatively low. There are no hazardous chemicals used in the processes that have to be discharged and there is no heat generation.

However, there are certain limitations to the application of membrane processes. A major disadvantage of membranes, especially in water- and wastewater-treatment processes, is that until today the long-term reliability has not completely been proven. Membrane processes sometimes require excessive pretreatment due to their sensitivity to concentration polarization, chemical interaction with water constituents, and fouling. Membranes are mechanically not very robust and can easily be destroyed by a malfunction in the operating procedure.

Another critical issure are the process costs. Generally, membrane processes are quite energy efficient. However, the energy consumption is only part of the total process costs. Other factors determining the overall economics of a process include the investment-related cost, which is determined by the cost of the membranes and other process equipment and their useful life under operating conditions, and various pre-and post-treatment procedures of the feed solutions and the products. Plant capacity may also play a role in total cost. While, for example, in distillation process usually a substantial cost reduction can be achieved with an increase in the plant capacity, the scale – up factor has only a relatively small effect on the process cost in desalination of water by reverse osmosis or electrodialysis.

Depending on the composition of the feed solution and the required quality of the product water, a combination of processes may be appropriate. For example, if ultrapure water for certain industrial applications is required, a sequence of processes may be applied, such as reverse osmosis, ion exchanger, UV sterilization, and microfiltration as a point-of-use-filter to remove traces of particles. The environmental impact of all membrane processes is relatively low. There are no hazardous chemicals used in membrane processes which have to be discharged. In certain applications such as the purification of industrial effluents and wastewaters or the desalination of brackish water there may be a problem with the disposal of the concentrate. In these applications brine post-treatment procedures may be necessary.

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DEFINITIONS

Back-flushing – A technique of pumping the permeate in the reverse direction through the membrane.

Fouling – The potential deposition and accumulation of constituents in the feed stream on the membrane . The deposition of material on the membrane surface and/or in its pores, leading to a change in the membrane performance.

Flux – Flow of physical property in space (mass and energy). Amount of permeate, or of any component in the permeate, that is transported through a membrane per unit of membrane area and per unit of time.

Hollow-fiber – Consists of a bundle of hundreds to thousands of hollow-fibers.

Membrane – A selective barrier that allows the passage of certain constituents and retains other constituents.

Permeability – Permeation of a gas or vapor through solid substance.

Permeate – The constituents that passes through the membrane.

Plate and Frame – A Membrane that consist of a series of flat membrane sheets and support plates.

Rejection – Amount of particles that have been removed from the feed.

Retentate – The constituents that containing the retained components.

Scaling – Formation of chemical precipitates in membrane.

Spiral-wound – A Membrane where a flexible permeate spacer is placed between two flat membranes sheet.

Tubular – Classification of membrane where the membrane are placed inside a support porous tubes and placed together in a cylindrical shell to form the unit module.

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Concentrate - The volume of liquid exiting a membrane system after flowing over the membrane, not through it. It is expressed as feed less permeate; it is also called brine, reject and retentate.

Feed - The volume of liquid entering a membrane system.

Housing - The part of the membrane filtration equipment holding spiral wound elements or other types of membrane elements, also referred to as pressure vessel.

Pressure drop - The loss of pressure between inlet and outlet of a membrane system, a housing (pressure vessel) or a membrane element.

Pressure osmotic - A theoretical value which highly impacts reality. The osmotic pressure is a function of the content of salt and other low MW solutes. High MW solutes can theoretically not contribute to osmotic pressure, but in reality a phenomena is experienced which resembles osmotic pressure. The osmotic pressure of electrolytes and low MW solutes can be calculated with some accuracy, but the "osmotic pressure" of high MW solutes can only be determined by experimentation.

Bubble point pressure - Pressure at which a continuous stream of gas bubbles is pressed through a liquid wet filter.

Bulk temperature - Temperature that exists in the bulk phase; in practice this temperature is equal to the measured temperature.

Module - The smallest practical unit containing one or more membranes and supporting structures (old terms to be replaced are permeator, membrane element).

Permeate - The portion of the feed passing through the membrane. Distillate can also be used as a term to describe the "permeate" of membrane distillation, but it is better to use "permeate" because it is commonly used in membrane literature.

Pore size - Openings in a membrane; this term is preferred to "pore diameter" and "pore radius", because all pore shapes can be described by this term.

Porosity - The porosity is defined as the volume of gas that is trapped inside a membrane divided by the total volume of the membrane;

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Retention - The ability of a membrane to hinder a component from passing through it or to retain a component in the fluid.

Retention coefficient - The degree of separation of a certain component from the solvent by the membrane under defined operating conditions;

Permeability - The property or condition of being permeable. The rate of flow of a liquid or gas through a porous material.

Diffusion-The passive movement of molecules or particles along a concentration gradient, or from regions of higher to regions of lower concentration. The spontaneous net movement of particles down their concentration gradient (i.e. difference in the concentrations of substances or molecules between two areas).

Reverse osmosis (RO) - a water purification technology that uses a semipermeable membrane. This membrane technology is not properly a filtration method. In reverse osmosis, an applied pressure is used to overcome osmotic pressure, a colligative property, that is driven by chemical potential, a thermodynamic parameter. Reverse osmosis can remove many types of molecules and ions from solutions, and is used in both industrial processes and the production of potable water.

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NOMENCLATURE

A a constant,

A_m membrane area,cm2;

A_w Solvent permeability constant (kg solvent/s m² atm)
A_s Solute permeability constant (kg solvent/s m² atm)

c Salt concentration (kg NaCl / kg H₂O)

B variable in liquid separation

c1 Solute concentration in feed (g solute/L)c2 Solute concentration in product (g solute/L)

cw2 the concentration of solvent in stream 2 (kg solvent/m³).

D Diffusion coefficent

D_i diffusivity of the solute I in the solution

D_{Ki} Knudsen diffusivity (cm²/s) = $4850D_p (T / M_i)^{1/2}$

Dp diameter pores (m) Dp pores diameter (cm)

F the total feed flow rate (cm3 (STP)/s) J_i The flux of component i (m³ / m²)

 J_s Salt Flux (m³ / m²)

 J_w A link to the pressure and concentration gradients across the membrane

K Henry's law sorption coefficient

K_f restrictive factor Mr Molecular weight

N_w Solvent flux (Kg solvent/s m²) N_s Solute flux (Kg solute/s m²)

P Constant of Membrane Permeability

P_{MA} Permeabilties component A, cm3(STP)/(s cm3 cm Hg)
P_{MB} Permeabilties component B, cm3(STP)/(s cm3 cm Hg)
p_h the total pressure in the high pressure (feed) side, cm Hg;

P_i Is a Constant of Membrane Permeability

 $p_{i(l)}$ Partial pressure of the component at the other side

 $p_{i(o)}$ Partial pressure of the component i

p₁ the total pressure in the low pressure or permeate side, cm Hg;

PMA the permeability of A in the membrane, cm3 (STP) cm/(s cm2 cm Hg);

q0 outlet reject flow rate (cm3 (STP)/s)

q_A the flow rate of A in permeate, cm3 (STP)/s; q_P outlet permeate flow rate (cm3 (STP)/s)

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R	Rejection	(%)

t Membrane thickness (m)

T temperature (K) v velocity rate (m³/m² s)

x₀ the mole fraction of A in the reject side; and

y_p the mole fraction of A in the permeate

Greek Leters

μ	Viscosity fluid (Pa s)
α	The membrane selectivity
α_{ij}	The membrane selectivity
3	Membrane porosity / volume fraction of pores in the membrane
Δр	the pressure difference across the membrane
Δπ	the osmotic pressure differential across the membrane(atm)
π1	Inlet osmotic pressure (atm)
π2	Outlet osmotic pressure (atm)
ρ1	Inlet density (kg/m³)
ρ2	Outlet density (kg/m³)
θ	Fraction permeated
Т	tortuosity

Superscript

А	a constant,
D	Diffusion coefficent
F	the total feed flow rate (cm3 (STP)/s)
K	Henry's law sorption coefficient
Р	Constant of Membrane Permeability
R	Rejection (%)

a constant

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