

1 An Introduction to Barrier Separation

1.1 Separation is ...

Separation is the key to the uses of nature. – Gathering, harvesting, mining are elementary manifestations of selection, typifying the objective of all separation, which is added value to the product procured.

Generic categories of separation are:

- Enrichment, enhancing the proportion of a target component;
- Isolation, recovering a target product from unwanted material;
- Extraction, same when employing a liquid extractant;
- Depletion, refers to the target product in the residue of isolation;
- Purification, removing impurities from the wanted product;
- Refining, purification in specific industries or circumstances;
- Fractionation, dividing into components or component groups;
- Phase separation, parting into mutually immiscible liquid phases;
- Precipitation, rendering a solution component insoluble;
- Volume reduction, concentrating dissolved species by removal of solvent;
- Dehydration, concentrating foods and biomass by removal of water.

Membranes are instrumental in many of these.

As to technical categories of separation, King [1] lists 54 separation processes in 11 categories. Different separation processes often are applied to the same separation task, the merits of one approach then having to be assessed in comparison to others. As *membrane processes*, barrier separations add to the inventory of separation science, showing specific advantages in some applications (for example hemodialysis; azeotrope splitting; bioseparations; ultrapure water; fuel cells), while competing on equal terms with traditional

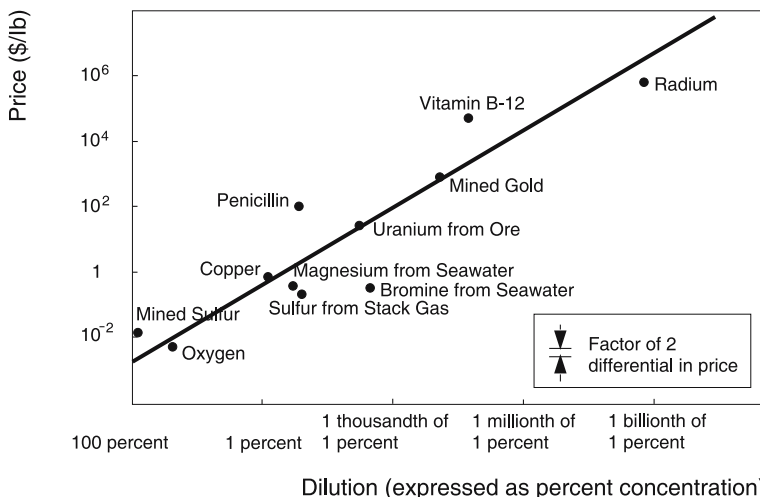


Fig. 1.1. The Sherwood plot: Selling prices of materials correlate with their degree of dilution in the initial matrix from which they are being separated. Taken from [2].

processes in many others. As a conspicuous example, membrane processes compete with distillation in water demineralization.

More often than not, separation is focused on the minority component(s) of mixtures: As wanted product to be recovered from a low-valued matrix or, conversely, as impurity to be removed to upgrade the matrix. In either mode, the expenditure to separate the minority component increases with dilution; dilution, in turn, increases with depletion. Specifically, recovery of valuable solutes from dilute liquid solutions is dominated by the cost of processing large masses of unwanted solvent. *Sherwood plots* illustrate a linear correlation between selling price of materials and their degree of dilution in the initial matrix when presented on a logarithmic scale (Fig. 1.1). Solvent removal from dilute solutions by *membrane filtration* effectively leads to solute enrichment, but just as well may serve as a means to purify the solvent.

The mechanism of separation is mass transfer. – Any mass transfer operation which produces a change in composition of a given feed mixture without permanently altering the identity of its components inherently is a separation. Any such operation yields – at

least – two product mixtures which differ in composition from one another and from the original feed. If one of the products is considered the target fraction of the separation, the other, by necessity, is the original feed devoid of the target fraction. The separation effect or *selectivity* of the process is assessed by comparing the analytical composition of the two products, or by relating the composition of either one of the products to that of the original feed. The objective of separation process design usually is to render one of the products as pure as possible.

Separation is demixing. – Selective mass transfer within a multi-component system enhances the degree of order, counteracting the natural tendency to uniform mixing, and thus requires energy. According to the thermodynamics of mixtures, the *minimum energy* to isolate a pure component species from a mixture or solution is proportional to $(-\ln x_i)$, where x_i is the mol fraction of that species in the feed mixture (Sect. 2.2.3). In terms of ordinary concentration, this proportionality is the reference coordinate of the *Sherwood plots* depicting cost of product recovery as function of initial product concentration. Conversely, the minimum energy to recover pure solvent from a given solution increases in proportion to solute concentration, affirming that the solute disturbs the thermodynamic condition of the solvent. Actual energy requirements may exceed the theoretical minimum by an order of magnitude, providing ample incentive for separation process development.

1.2 Barrier separation is ...

Barrier separation is rate controlled mass transfer. – Barrier separations rely on mass transport across *semipermeable* physical partitions, selectivity coming about by differences in *permeability* of the barrier towards the feed components resulting in the rates of mass transfer to differ. The operative distinction of rate governed versus equilibrium separation is dynamics: Mass transfer through a barrier is slowed by molecular interaction with the barrier matrix (figuratively viewed as *friction* on a molecular level), and likely is affected by encounters between the permeating species en route (loosely referred to as *coupling*); this is the essence of *barrier interference*. By comparison, mass transfer across a liquid-vapor interface (VLE

= vapor-liquid equilibrium) is considered instantaneous, and interaction between the vaporized species is negligible.

The two generic products of barrier separation are the *permeate* (= the fraction transported through the barrier), and the *retentate* (= the fraction retained or rejected by the barrier), Fig. 1.2. Although either one may be the target fraction of the process, analysis of barrier separation is by relating the permeating fraction to the feed, thereby registering the influences of barrier interference and process conditions. Feed components present within the barrier at any time are the *permeants* (*penetrants* to some).

The term *semipermeable membrane* was introduced by van't Hoff (1887) [3], originally denoting an ideal barrier permeable to solvent (water) only while being completely impermeable to dissolved species (Sect. 3.1.2). Such a membrane would stabilize the osmotic equilibrium between a liquid solution and its own pure solvent. The contrary limit is a freely permeable, nonselective barrier yielding a permeate identical in composition to that of the feed, – in effect a throttle. Real barriers, even though selected or designed for high selectivity, are “leaky” in that, in principle, they are permeable to all species encountered. The ultimate state of a system of fluid mixtures in contact with any real membrane would be complete uniform mixing, if only one would wait long enough. There is thus no absolute barrier separation on two counts: The process is self-quenching, the energy to remove the selectively permeating species increasing with depletion; and, real membranes are leaky.

The earliest barrier on record is a section of moist pig's bladder stretched over the mouth of medicine bottles before cork stoppers came into use, hence the terms *membrane* and, in due course, *mem-*

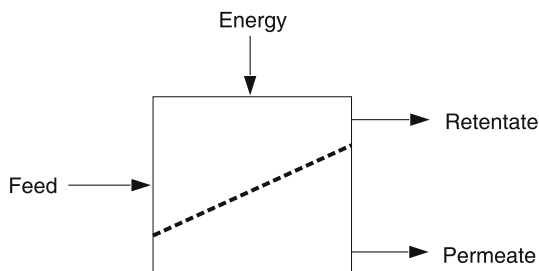


Fig. 1.2. Pictograph of a barrier separation stage.

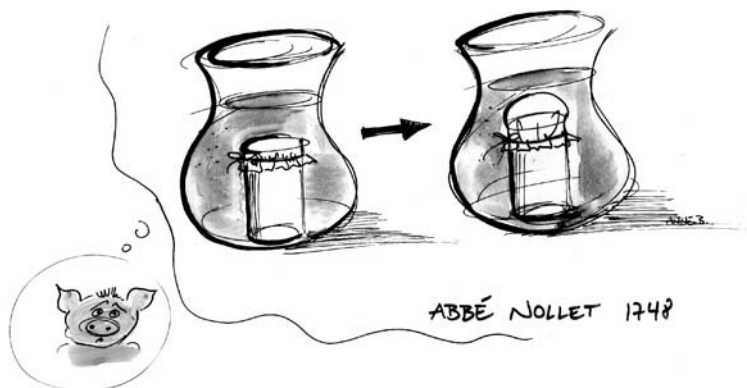


Fig. 1.3. A sketchbook impression of Nollet's chance discovery of semipermeability: Water entering a membrane-capped vial containing "spirit of wine" creates pressure (courtesy Anne Bøddeker).

brane process. Selective permeability came as a surprise to Nollet (1748) when he discovered that pig's bladder is more permeable to water than to "spirit of wine" (ethanol), resulting in a pressure phenomenon seemingly out of nowhere (Chapter 7).

1.3 Membranes, economy of size and affinity

Membranes are defined by what they do, rather than what they are. – Nature and man's ingenuity provide an abundant variety of barrier materials, both organic and inorganic, having the capacity of being permeable to individual fluids (liquids, vapors, gases), and semipermeable (selectively permeable) to fluid mixtures (Appendices D and E). The models describing membrane mass transport seek to relate *structure* and *function* of the barriers, reducing the material variety to a few phenotypes as follows.

Porous barriers, operating on size discrimination, conform to the notion of "filters": The solvent moves more or less freely, dissolved species are discriminated upon. The criterion distinguishing *membrane filtration* from ordinary (particle) filtration is solute size, smaller solutes requiring narrower pores to be retained. While gravity is all it needs to drive ordinary filtration, narrow pores require

a pressure head to overcome the *hydraulic resistance* of the pore structure; the *filtration spectrum* of pressure versus solute diameter, shown in Fig. 4.2, covers the operative range of *membrane filtration*. Since specific solute species tend to be more uniform in size (respectively mass) than is met by the *pore size distribution* of most porous membrane materials, membrane characterization is in terms of rejection functions with respect to given solute size (Sect. 4.4). A pore size commensurate with a solute diameter of $0.2\ \mu\text{m}$ is noteworthy in that it nominally excludes bacteria from water by *microfiltration*.

Gaseous diffusion through porous (inorganic or metallic) barriers follows a different mechanism, being governed by considerations of pore geometry versus mean free path (= pressure) of the gas or gaseous mixture components.

A survey of microporous structures is presented in Appendix E.

Homogeneous barriers (nonporous or “dense”) discriminate according to relative solubilities and diffusivities of the feed components in the membrane phase. Unlike porous barriers, *solution-diffusion* type barriers rely on specific interactions of the permeants with the membrane material, its chemistry and molecular morphology. With a view at performance, more than on principle, membrane polymers (Appendix D) are assigned to one or both of the following categories,

- as *glassy* (crystalline) versus *rubbery* (elastomeric) by physical nature,
- as *hydrophilic* versus *hydrophobic* by interactive preference.

Attempting for guidance in diversity, glassy polymers generally show lower permeability and higher selectivity than rubbery ones. Liquid (aqueous-organic) separations are dominated by the sorption capacity of the membrane polymers, attended by swelling. By sorption preference, glassy polymers are hydrophilic, responding to water as being the smallest of liquid molecules at room temperature, whereas rubbery polymers tend to be organophilic (Sect. 6.2).

In practical gas separation, sorption of gases into polymers being low, the higher diffusive selectivity of glassy polymers outweighs the higher permeability of rubbery ones.

Liquid membranes function as solution-diffusion barriers, providing the very high diffusivity to permeants characteristic of the liquid state. Consequently, selective mass transport is expected to be

governed by the rules of solute distribution (partition) between immiscible liquid phases in contact. *Facilitated transport* makes use of mobile carriers incorporated in the liquid membrane phase to provide species-specific selectivity.

Functionalized membranes, adding chemistry to polymer science, attempt to modify the barrier as a whole or the barrier surface to facilitate selective sorption, or else to counteract undesired membrane fouling by chemical means. A prime objective is to convey hydrophilicity to membranes used in aqueous separations, notably to reduce fouling by proteins. Another objective is resistivity towards oxidizing agents, chlorine in particular, widely employed to disinfect feed waters in water treatment.

A category of functionalized membranes of their own are charged membranes coming as anion exchangers (positive fixed charges) and cation exchangers (negative fixed charges). As “immobilized electrolytes”, charged membranes are anticipated to be highly hydrophilic. When employed in *electrodialysis*, mass transport, pertaining to charged species only, is by combined action of *ionic conduction* and *Donnan exclusion* under the driving force of an electric potential.

The role of water. Water is a key component in liquid barrier separation, as is water vapor in gas separation. Not surprisingly, the presence of water within a membrane is a telltale piece of information on the nature of that membrane. With reference to the above phenotypes:

- Water in porous barriers is pore fluid. Indeed, as long as water sorption by the membrane (polymer) material itself is negligible, the difference in weight between “wet” and “dry” should equal the void space within the membrane structure (then termed *volume porosity* as against *surface porosity*, Sect. 4.2). Mass transport of solutes smaller than pore dimension is by *convection* (as in *membrane filtration*) and/or by *diffusion* within the pore fluid (as in *dialysis*). Even though mass transport is confined to the pores, the nature of the polymer matrix does matter. For example, a hydrophobic porous barrier like a microporous PTFE (*Teflon*) membrane may prevent liquid water to enter but will allow water vapor to pass (as in *membrane distillation* – and breathable textiles).
- Water absorbed (dissolved) by homogeneous polymers may be considered as a molecular solute in a polymeric solvent, causing

the polymer to swell. Sorption capacity depends on the relevant interactive forces (*hydrogen bonds* and *polarity*, Sect. 6.3), but also on the “stiffness” of the polymer matrix (*glassy* versus *rubbery*) resisting polymer swelling. As an orientational aid, the dense salt rejecting layer of a composite hydrophilic membrane as employed in water desalination by *reverse osmosis* typically contains 10% of dissolved water, whereas the porous support of such a membrane may have a “porosity” (water as pore fluid) exceeding 60%.

- Charged polymers (ion exchange membranes), by both their fixed charges and mobile counter ions, provide ample ion-dipole attraction for water storage. With up to 30% of water their consistency is that of a swollen gel with restricted water mobility. However, when modeling solute mass transfer (as of ions in *electrodialysis*), ion exchange membranes are pictured as porous with the charges lining the pore walls.

1.4 Driving force, actuating barrier interference

Maxwell's demon needs help. – Next to the membranes, agents of barrier separation are the operating conditions which provide the driving force for selective mass transport

- against the inherent resistance of any mixture to demixing (this is where Maxwell's demon comes in);
- against the cohesive energy of fluid mixtures (this is where molecular interaction comes in);
- against the dynamic (transport) resistance of the barrier (this is where barrier interference comes in).

In form of the respective *gradients*, the driving force is composed of the very same variables which describe the thermodynamic condition of the fluid mixtures contacting the membrane, – *temperature*, *pressure*, and *composition*. Between them, these *intensive properties* (independent of total mass) constitute the *Gibbs free energy* or *free enthalpy* of the mixture (G). The free energy of any individual mixture component, its *partial molar free energy*, after Gibbs is named the *chemical potential* of that component species within the mixture

(μ_i). It becomes manifest as change in free energy of the mixture as the concentration of the component under consideration varies, as, for example, upon its removal in a separation process.

In actual practice, there is no need to explicitly include a temperature gradient among the driving forces since barrier separations for the most part are isothermal, usually operating at ambient (including bio-ambient) temperature. A case of exception is *membrane distillation*, which requires a thermal gradient across the porous barrier. – Likewise, an *electrochemical potential* is not included in the general treatment, electromembrane processes being confined to a class entirely of their own [12]. – On the whole, therefore, the relevant driving forces in barrier separation derive from *pressure* and *composition* of the fluid mixtures to be separated.

Pressure is the “natural” driving potential in all filtration operations, which are characterized by preferential transport of solvent (water) over solute, hence the nominal inclusion of *reverse osmosis* as “hyperfiltration” (the common expression “desalination by reverse osmosis” is misleading, “dewatering” is called forth). The upper reach of pressure encountered in membrane filtration is 100 bar (10 MPa); at this pressure ordinary liquids are incompressible, however, porous or swollen polymers are not, neither are microorganisms. – *Gas permeation* through, and *gas separation* by, homogeneous polymer membranes likewise is pressure driven, as is *gaseous diffusion* across microporous barriers.

Akin to pressure, **vapor pressure** is a driving force in barrier separation. Depending on how the vapor pressure gradient is created, the relevant membrane processes are:

- *Membrane distillation*, the only membrane process operating on a temperature gradient between liquid feed and liquid permeate. The membrane is a porous hydrophobic (water-repellent) barrier permeable to water vapor only; water transport is by evaporation into the pore space followed by re-condensation on the permeate side. – In *osmotic distillation* the vapor pressure gradient is created, not by temperature, but by a difference in solute concentration, a high solute concentration creating a vapor pressure “sink” on the permeate side, irrespective of the nature of the solute used (Sect. 2.1.1). Gentle dehydration is the usual objective of both process variants.

- *Pervaporation* is a hybrid, operating on a drastic reduction of vapor pressure (of partial pressures in case of volatile mixtures) by causing the permeants to evaporate as they emerge from the membrane. In effect, pervaporation may be viewed as nonequilibrium vacuum distillation across interacting (solution-diffusion type) barriers, usually applied to “difficult” liquid separations: Separation of narrow boiling or constant boiling (azeotropic) mixtures; separation of high boiling organics from aqueous solution (Sect. 5.3).

Composition. While pressure as driving force for mass transport conforms to intuition, *concentration gradients* do not. In fact, nature’s urge to establish and maintain uniform mixing within fluid mixtures at all cost represents a powerful driving force for mass movement. It is a *virtual force*, it is the motor of *diffusion*. If, given a concentration imbalance, diffusive mixing is intercepted by a permeable barrier, mass flows will adjust themselves predictably to the permeability situation:

- A porous membrane will allow “small” solute species (including the solvent itself) to equilibrate more or less freely while retaining macromolecules. This is the operating principle of *dialysis*, hemodialysis as an example. – *Electrodialysis* is a namesake in that it, too, relocates the solute.
- With a homogeneous (“dense”) membrane, if at all permeable to solvent (water), there is only one way to comply with nature’s call to mitigate concentration differences: By allowing water to cross from the dilute to the concentrated side of the membrane. This is the phenomenon of *osmosis* (Sect. 3.1.2).

1.5 Dynamics of barrier separation

Mass transport is molecular motion with a directional bias. – It is slow motion, as a simple calculation will illustrate: At a throughput (*flux*) of 1000 L/d m^2 (low for ultrafiltration, high for reverse osmosis) the apparent linear velocity of mass transport within the membrane is about 4 cm/h or little more than 10^{-3} cm/s . To be sure, except for revealing a net relocation, this is no information on the

actual random motion of the permeants in the membrane phase (which is a subject of *molecular modeling*).

Performance. – The formal relation between mass flux and driving force has the structure of a *generalized Ohmic law*: Flux is proportional to driving force. The coefficient of proportionality (a reciprocal resistance in the Ohmic analogy) has two meanings depending on how the driving force is introduced:

- It is a *permeability* when flux follows a *gradient* of the potential; by confining the gradient to within the membrane boundaries (“difference approximation”, Sect. 2.2.2), membrane thickness becomes part of the permeability format.
- It is a *permeance* when, for a given membrane, the causality between observed flux and applied potential (as pressure or individual feed concentration) matters; it is thereby a record of performance.

$$\text{Flux} = \text{Permeability} \times \text{Potential gradient}$$

$$\text{Flux} = \text{Permeance} \times \text{Potential}$$

Permeability characterizes the transport capability of the barrier material itself; it thus allows for membrane material evaluation. The permeance of a given membrane (sometimes called its “productivity”) is the experimentally observed flux as function of operating conditions (see Figs. 3.3; 4.4; 5.3; 5.6). If the thickness of the membrane is known, permeability and permeance correspond, permeability appearing as thickness-normalized permeance.

Barrier separations coming about through differences in transport rate of the permeants, the ratio of individual permeabilities (or permeances) suggests itself as a measure of the separation effect:

$$\text{Selectivity } (ij) = \text{Permeability } i \text{ (high)} / \text{Permeability } j \text{ (low)}$$

While this relation is formally correct, it is no recipe to estimate, much less to predict practical membrane separations, for two reasons: Individual (single component) permeabilities often are inaccessible (imagine pure salt permeability); if they are, their numerical ratio misjudges the interactions which make barrier separations interesting. It is only with true (“permanent”) gases that the ratio of pure component permeabilities, individually established, quantitatively predicts the separation effect (then referred to as *ideal separation*).

Nevertheless, where accessible, single component permeability (or permance) provides information on the intrinsic transport behavior of the barrier; pure water permeability of microporous membranes, in particular, is a key criterion in *membrane filtration*.

Concentration polarization. – The most influential effect of process dynamics on rate-governed separations by far. Referring to a gradient in composition within the feed phase next to the membrane surface, concentration polarization is a consequence of the slower permeating feed component accumulating near the solution-membrane interface as the faster permeating component moves on. As a result, the feed mixture as “seen” by the membrane differs in composition from the bulk feed, aggravating the separation task. If it is the *solvent* to permeate preferentially (as in *reverse osmosis* and all *membrane filtrations*), the *solute* being retained, concentration polarization requires conditions to be adjusted to a higher than bulk solute concentration. Conversely, if the solute or minority component permeates preferentially (as in *pervaporation* and *dialysis*), solute depletion near the membrane boundary effectively causes a lower than bulk concentration. It is to alleviate these effects that barrier separations almost always operate in the *cross flow* (tangential flow) mode, to be contrasted with *dead end* filtration.

Concentration polarization is a phenomenon to be reckoned with in liquid barrier separations. In the limit of *perfect mixing* of the feed components, as is generally the case when handling gas mixtures, the effect is irrelevant.

Whereas concentration polarization is a boundary layer effect readily rationalized, the mutual influence of permeating species on their transport behavior, referred to as *coupling*, is not easily predicted and needs case by case attention. By tendency, coupling would be expected to impair selectivity by leveling differences in mobility of the permeants, – reminiscent of the individual freedom of ions in solution being restricted by the condition of electroneutrality.

1.6 On units and dimensions

Permeability and *selectivity* are categories of performance rather than units by themselves. Reduction to practical needs is by identifying the parameters involved, both by their physical meaning and

by dimension, then assigning appropriate units to the parameters identified.

It is noted that true SI units (the system dating back to 1960), besides not being universally accepted, rarely answer the needs of practical separation processing. Examples for unwieldy SI units are: Pascal (Pa) for pressure [replaced in this text by bar; $1 \text{ bar} = 10^5 \text{ Pa} = 0.1 \text{ MPa}$]; second (s) for time [in most cases replaced by hour (h) or day (d)]. Both kg (for “mass”) and mol (for “amount of substance”) are SI base units; yet, a mol of a specified substance is still a *mass* to be expressed in kg/mol . As an aside it is observed that industrial output is not normally reported in *mols of product*, – and if so, it would have to be *number of mols* (n) which, when multiplied with the respective molecular weight, is a true mass again (kg).

In the following some key parameters of barrier separation are discussed, using SI base units and hinting at SI derived units. It is noted that *volume*, a preeminent parameter in fluid mass transfer, is not a base unit in the SI system, although m^3 and L (liter) belong as SI derived.

Flux (J) [$kg/s\ m^2$] or [$mol/s\ m^2$]. – Flux is the quantity of permeant collected in a time (flow rate) at given membrane area, hence a *flow density* by dimension. Total flux in multiple component permeation is the sum of individual fluxes, established retrospectively by analyzing the permeate composition. Adaption to practical units, including to volume flux, is self-evident; for example, the common flux unit [$L/h\ m^2$] passes as SI derived. Dimensionally reducing a *volume flux* to a *velocity* [$m^3/s\ m^2 \rightarrow m/s$], except for implementing *mass transfer coefficients* (Sect. 4.2), in most cases distracts from the physical meaning of the compound unit.

Permeability (L). – Permeability has many faces, all of the same dimensional configuration: Flux as function of driving force.

- The driving force for each component is a *gradient* of its chemical potential in terms of pressure or concentration (Sect. 1.4), hence the SI unit [$kg\ m/s\ m^2\ bar$] when considering pressure-driven processes.
- Phenomenologically, permeability covers the sequence of events as a permeating mixture component makes its way from bulk feed into membrane (sorption) and thence across the membrane (diffusion), boundary layer influences and coupling effects inclusive;

it is thus a record of barrier interference. – Gas permeation is characterized by a low level of molecular interaction; individual gas permeabilities are still recorded in *Barrer units* as a semi-standard (using “cmHg” for pressure).

- Liquid permeation through porous membranes (as in membrane filtration) is described as *hydraulic permeability* (L_p); it is convective – as opposed to diffusive – volume flux (J_v) driven by a hydraulic pressure gradient [bar/m]. Pure water hydraulic permeability is one of the parameters characterizing a porous membrane. Analysis of hydraulic permeability, true to the Ohmic law analogy, is in terms of the *resistance* of the barrier to liquid transport; solute deposited on the membrane surface adds to the overall resistance (gel polarization, Sect. 4.2.2).

Permeance. – Rather than to a potential *gradient*, permeance relates the flux to the potential itself, to pressure or concentration of the permeating species. When referring to a constant pressure as driving force, permeance appears as *pressure-normalized flux*, [kg/s m² bar] in SI units. *Concentration-normalized flux* (having the dimension of a *mass transfer coefficient*), besides applying to controlled laboratory conditions, refers to separations at constant composition feed supply (seawater, for example). In batch operation, which is identical to plant operation under conditions of recovery, there is a methodical concentration dependence of flux instead (Sect. 2.2.2). – A decidedly non-SI unit of permeance is the concoc-tion [gfd/psi], encountered in water treatment (refer to list of abbreviations).

Since sorption is prerequisite to solution-diffusion governed mass transfer, a correspondence between permeance and sorption isotherms (Sect. 2.3.1) is anticipated.

Selectivity. – Selectivity is a statement of separation performance based on a comparison of analytical compositions of feed (“bulk”) and permeate. Practical needs dictate which form is used to express selectivity (Sect. 5.2 has examples). *Intrinsic selectivity* refers to the true separation capability of the barrier under undisturbed conditions, – absence of concentration polarization in particular.

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