MEMBRANES IN SEPARATIONS

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Chapter III

MECHANISMS OF MEMBRANE TRANSPORT

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The mechanisms involved in the variety of possible membrane processes fall into categories of basic transport phenomena.

Thus, the scope of this chapter is a presentation of definitions for the specific processes and the delineation of appropriate mass-transfer models. This should clarify the involved processes and provide pertinent distinctions, for instance, between reverse osmosis and ultrafiltration.
\[ N_i = J_i^* = - D \frac{\partial C_i}{\partial x} \]  

(3.8)

This is the equation that most people use in membrane permeation. They interpret the diffusional flux as being the permeation flux. Even though this may be a good approximation for many systems, we now clearly know that Eq. 3.8 is not valid in general. The error involved in this approximation is the second term of Eq. 3.6, that is, \( C_i \nu^* \). When the permeation flux is very large, this may not be negligible.

To illustrate this point, let us consider a single-component system. There is only one species A permeating through the membrane. However, the membrane will act as a second component in our diffusion system. Using the subscript \( M \) for the membrane, Eq. 3.6 becomes

\[ J_A^* = N_A - C_A \nu^* = N_A - \frac{C_A}{C} (N_A + N_M); \]  

(3.9)

since the membrane does not move,

\[ N_M = 0. \]  

(3.10)

Then, Eq. 3.9 is reduced to

\[ J_A^* = \left(1 - \frac{C_A}{C}\right) N_A \]  

(3.11)

or

\[ N_A = \frac{C}{C_M} J_A^* = - \frac{C}{C_M} D \frac{\partial C_A}{\partial x}. \]  

(3.12)

Re-expressing Eq. 3.12 yields

\[ N_A = - D \frac{\partial C_A}{\partial x} - \frac{D}{2C_M} \frac{\partial C_A^2}{\partial x}. \]  

(3.13)

The permeation flux of A observed in the laboratory is \( N_A \), not \( J_A^* \). Thus in the general case any permeation data obtained experimentally should be correlated by using Eq. 3.13 and not Eq. 3.8.

The difference between Eq. 3.13 and Eq. 3.8 is the second term in Eq. 3.13, which may be quite small in many systems. This term is second order with respect to \( C_A \). Thus, we may expect, in general, a nonlinear relationship between the observed permeation flux and the overall concentration gradient for membrane processes. An important point is that the system still obeys so-called Fickian diffusion.
Similarly, Fick's second law should be re-examined for membrane systems. A material balance for component A gives

$$\frac{\partial C_A}{\partial t} + \frac{\partial N_A}{\partial x} = R_A,$$  \hspace{1cm} (3.14)

where $R_A$ is the molar rate of A produced per unit volume of the system due to chemical reaction. Substituting Eq. 3.13 into 3.14 for a system with no chemical reaction yields

$$\frac{\partial C_A}{\partial t} = \frac{\partial}{\partial x} \left( D \frac{\partial C_A}{\partial x} \right) + \frac{\partial}{\partial x} \left( D \frac{C_A}{C_M} \frac{\partial C_A}{\partial x} \right).$$  \hspace{1cm} (3.15)

Comparing this equation with Eq. 3.2, we note that the new term, which is nonlinear with respect to $C_A$, appears in our modified second law of diffusion. It is still a Fickian type of diffusion. These modifications were necessary because the permeation flux $N_i$, which is the quantity measured in the laboratory, is not the diffusion flux $J_i^*$. The material flux going through a membrane should never be confused with the true diffusional flux.

The significance of the preceding treatment is as follows:

For membrane diffusion processes of all types, the correct transport equations are Eqs. 3.13 and 3.15. They differ from Fick's basic diffusion equations 3.1 and 3.2 in that second terms are added that are due to the presence of the membrane. In many instances, particularly in gaseous diffusion processes, the effect of the second term may be negligible.

2 HYDRODYNAMIC FLOW

When membranes are porous and the fluid flow is laminar, a simple hydrodynamic theory applies. In the case of gas-phase flow, the pores must be large enough compared with the mean free path of gas molecules to ensure viscous flow. For many membranes, the structures of pores are very complicated and frequently not known. Various models have been used with different degrees of success. Numerous pore-structure factors are introduced, such as average pore diameter, porosity, equivalent hydraulic radius, average length of capillary, particle diameter, pore size distribution, tortuosity, and specific surface area. We shall begin with the phenomenological discussions of hydrodynamic flow.

Darcy's Law

This basic law governing the flow of fluid through porous media was originally developed by Darcy [4]. It states that the flow rate is directly proportional to the pressure gradient causing flow. The linear relationship is once again an
example of the general phenomenological equations of fluxes and forces in nonequilibrium thermodynamics (see Eq. 2.16), so that

$$\frac{V}{St} = \frac{K}{\mu} \frac{\Delta P}{l}.$$  \hspace{1cm} (3.16)

Here the flow coefficient is divided by the viscosity of the fluid, and \( K \) is called the permeability. This definition of permeability differs from our earlier definition given by Eqs. 2.1 and 2.2. However, this practice is widespread in petroleum technology and in soil science.

The viscosity of a fluid is a measure of internal friction between fluid laminae flowing at different velocities. If a fluid undergoes laminar flow, this friction yields shear forces. When a flowing fluid contacts a solid surface, it adheres to the surface, resulting in a zero fluid velocity. As a consequence of the viscosity and the adhering property of the fluid, the solid surface experiences a drag force. The viscous resistance is a counter force to this drag.

Darcy's law expresses that the flow resistance is due to viscous drag, and the permeability \( K \) contains all the properties of the porous medium. This definition of permeability is intended to separate the fluid property, that is, the viscosity, from the pore-structure properties in the overall flow coefficient. The unit of Darcy's permeability is a darcy, which is a flow of 1 cc/(sec)(cm\(^2\)) with a pressure gradient of 1 atm/(cm) for a fluid with 1 cP viscosity. The explicit expression of permeability as a function of pure-structure parameters will depend on the specific pure models.

**Capillary Model (Hagen-Poiseuille Equation)**

When a porous membrane consists of straight cylindrical capillaries of equal size, the Hagen-Poiseuille equation should apply directly to describe the flow rate, and

$$\frac{V}{t} = \frac{nS\pi r^4}{8\mu} \frac{\Delta P}{l}.$$  \hspace{1cm} (3.17)

In terms of molar units:

$$F = \frac{V}{t} \frac{P}{RT} = \frac{nS\pi r^4 P}{8\mu RT} \frac{\Delta P}{l}.$$  \hspace{1cm} (3.18)

Comparing Eq. 3.16 and Eq. 3.17, we can obtain the Darcy permeability for the capillary model. Noting the porosity for the capillary membrane to be \( \varepsilon = n\pi r^2 \), the permeability becomes
\[ c_{\text{Na}^+}^{\text{I}} \cdot c_{\text{Cl}^-}^{\text{II}} = c_{\text{Na}^+}^{\text{II}} \cdot c_{\text{Cl}^-}^{\text{II}} \]  \quad (3.31)

The maintenance of electroneutrality is also required so that
\[ c_{\text{Na}^+}^{\text{I}} = c_{\text{Cl}^-}^{\text{I}} + c_{\text{X}^-}^{\text{I}} \]  \quad (3.32)
\[ c_{\text{Na}^+}^{\text{II}} = c_{\text{Cl}^-}^{\text{II}} \]  \quad (3.33)

Combining the above equations, the Donnan ratio can be obtained:
\[ \left( \frac{c_{\text{NaCl}}^{\text{II}}}{c_{\text{NaCl}}^{\text{I}}} \right)^2 = 1 + \frac{c_{\text{NaX}}^{\text{I}}}{c_{\text{NaCl}}^{\text{I}}} \]  \quad (3.34)

From this equation, it is obvious that an unequal distribution of the diffusible salt is achieved at the equilibrium state. The ratio of the NaCl concentrations in the two compartments is always greater than 1 because the concentrations cannot be negative.

12 KNUDSEN FLOW

The viscous flow governed by the Poiseuille formula is limited in the pressures and temperatures such that the mean free path of fluid molecules is very small compared to the diameter of the pores. If the mean free path becomes comparable or larger than the pore size, the fundamental concept of viscous flow, that is, the continuum concept, breaks down. In the continuum regime, the collision frequency between gas molecules exceeds greatly that of gas to wall. However, when the mean free path is much larger than the pore diameter, the collisions between gas molecules become much fewer than the collisions between gas molecules and the wall. This situation is known as a rarefied gas, and the flow in this limit is usually referred to as "free-molecule diffusion" or "Knudsen flow."

The original work of Knudsen in 1909 [30] contains his theory of free-molecule diffusion as well as experimental data of several gases. A typical example is given in Fig. 3.2 for the flow of CO₂ through a glass capillary (Capillary No. 4). In the high-pressure range the flow obeys the Poiseuille equation very closely. As the pressure becomes lower, the data points deviate from the straight line. After going through a minimum point, further lowering
of pressure causes the flow to increase slightly until it approaches asymptotically a limiting value. In the extremely low pressure range, the flow becomes independent of pressure, which is the true Knudsen regime. In the intermediate pressure region, no satisfactory theory can explain the flow data. Based on the kinetic theory of gases, Knudsen derived the following equation for free-molecule flow through a long circular capillary:

$$ F = \frac{8\pi^3}{3(2\pi RMT)^{1/2}} \frac{P_1 - P_2}{l} $$  \hspace{1cm} (3.35)

where $r$ is the radius of the capillary and $M$ is the molecular weight of the gas. Basically, the same equation applies to all microporous membranes with a little modification of a geometric factor $G$.

$$ F = \frac{GS}{(2\pi RMT)^{1/2}} \frac{P_1 - P_2}{l} $$  \hspace{1cm} (3.36)

Many authors have attempted to express the geometric factor $G$ in terms of more explicit quantities, such as tortuosity and porosity, based on different pore models.

For short capillaries, which can be considered as the model of actual porous materials, Clausing [31] developed the methods of calculating the flow as a function of the ratio of capillary length over capillary radius. At the limit of zero length, or a hole in an infinitely thin membrane, the flow equation is reduced to
\[ F = \frac{S}{(2\pi RMT)^{1/2}} (P_1 - P_2). \]  

This limiting flow is known as molecular effusion or Knudsen effusion. The above equation is simply the difference between two impinging molecular beams from both sides of the membrane through a cross section of \( S \).

Pollard and Present [32] also performed similar computations, but they extended their theory to the transition region, where the mean free path is comparable to the pore diameter. Their theory explains the minimum occurrence in the flow rate for a long capillary and its absence for a short capillary and for porous media. This fact has been well established experimentally for a long time. In the intermediate pressure range, the so-called "slip" flow is added customarily to the Poiseuille formula, so that essentially the Poiseuille equation can still be used with a slight modification. However, it should be pointed out that this approach is not rigorous.

There are a number of qualitative differences between Poiseuille flow and Knudsen flow. The Poiseuille formula, Eq. 3.18, contains the fourth power of the radius whereas the Knudsen equation, Eq. 3.35, contains the cube of the radius. The Poiseuille flow is inversely proportional to the viscosity of the fluid; however, the Knudsen equation does not involve the viscosity because there is no concept of viscosity in the rarefied gas (or Knudsen regime). The viscous flow increases linearly as the pressure drop increases: this point is identical with Knudsen flow; however, the Poiseuille equation is also dependent upon the average pressure whereas the Knudsen flow is not. When a mixture of gases flows through a tube according to the Poiseuille equation, each component cannot behave independently because of the molecular chaos. Therefore, the flow will be as if one kind of gas is going through the capillary with the average viscosity. There is never a separation. However, in the Knudsen regime, the component gases will flow through the capillary independently of each other. Thus, a separation takes place due to the difference in their molecular weight. This is the principle involved in the isotope separation of \( ^{235}\text{U} \) by means of the gaseous diffusion process.

In porous media, when the flow is in the free-molecule regime, the surface diffusion is also present. The relative magnitude of surface flow with respect to the total flow is determined by many factors. A detailed discussion will follow in a later chapter.

13 THERMAL EFFECTS

So far our discussions have been limited to the phenomena taking place in isothermal systems. Imposing a temperature gradient across membranes, we can also expect a mass-transfer process based on the theory of nonequilibrium...
thermodynamics, Eq. 2.16. When the temperature gradient causes diffusion, this process is known as thermal diffusion or the "Soret effect." If the temperature gradient is responsible for the flow of solvent, it is called thermooosmosis. Yet, if the flow is in the Knudsen regime, it is called thermal effusion.

Denbigh [33] was the first to demonstrate experimentally that either CO$_2$ or H$_2$ passes through a nonporous rubber membrane under a temperature gradient. The theoretical interpretation of this coupling phenomenon is well presented by Katchalsky [34]. In the liquid phase, Rastogi and co-workers [35] reported measurements of the thermo-osmotic permeability of a Du Pont 600 cellophane membrane using water. However, Carr and Sollner [36] found no transport of water through a collodion membrane. A speculative discussion was presented by Spanner [37] on the significance of thermo-osmosis for biological membranes.

Thermal effusion is a much more established phenomenon, both theoretically and experimentally. Since it is rarefied gas flow, the effusion equation can be obtained from Eq. 3.37 as

$$F = \frac{S}{(2\pi RM)^{1/2}} \left( \frac{P_1}{T_1^{1/2}} - \frac{P_2}{T_2^{1/2}} \right). \tag{3.38}$$

At equilibrium, when there is no flow, the pressures at both sides of the membrane are related by

$$\frac{P_1}{P_2} = \left( \frac{T_1}{T_2} \right)^{1/2}. \tag{3.39}$$

As in the case of Knudsen flow for an isothermal system, a separation is also possible for two different gases if their molecular weights are different.

The above thermal phenomena do not play a significant role in any membrane-separation process, since the magnitudes of the coupled phenomena are usually quite small compared to the conjugated phenomena (principal phenomena). However, they may have some applications in an unusual environment.

14 FACILITATED DIFFUSION

All the phenomena hitherto described have been pure mass-transport processes under a concentration or pressure gradient. When the simple diffusion process is coupled with chemical reactions, the net transport rate may be greatly affected. Usually, it is facilitated. Thus, the name facilitated diffusion is given to a system in which the rate of transport is increased from the expected value based on the simple diffusion theory alone. However, facilitated diffusion cannot change the final equilibrium that may be reached by simple diffusion.
It only speeds up the rate of attainment of the equilibrium. Another characteristic feature of this facilitated diffusion is the fact that the rate of permeation increases proportionally with increase of concentration gradient only to a limit, beyond which it varies little with further increase of the driving force. This limiting permeation rate suggests several possible mechanisms. A "carrier" system is one, and an augmenting chemical reaction is the other.

The carrier model assumes a carrier substance inside the membrane that shuttles back and forth between the two interfaces carrying the permeant molecules. A good example of this model would be the oxygen transport via hemoglobin molecules studied by Scholander [38]. He observed more than eightfold increase in oxygen transfer rate when hemoglobin molecules are present. This facilitated diffusion was explained as being based on the concept of a hemoglobin carrier model. The oxygen molecules are transmitted from one hemoglobin molecule to the next in a chain. As the pressure of air was changed, the rate of nitrogen diffusion increased proportionally, but the oxygen-transport rate remained constant over a wide pressure range.

The second model assumes chemical reactions that are taking place inside the membrane, and the reaction products actively participate in the diffusion process. A simplified version of this model will be given here for a binary system. A membrane containing a suitable solution divides the system into two parts. One side of the membrane is filled with species A and the other with species B initially. Both A and B will diffuse through the membrane according to their own concentration gradients. Inside the membrane, A and B react to form AB, which will also diffuse within the membrane due to its own concentration gradient:

\[ A + B \xrightarrow{\frac{k_1}{k_2}} AB . \]  

(3.40)

The schematic picture is shown in Fig. 3.3. If the equilibrium conditions for the above reaction are such that the concentration gradient of the product AB is in the same direction as the concentration gradient of A, the diffusion of AB will take place in the same direction as the diffusion of A. When the product AB reaches the other side of the membrane interface, it will decompose into A and B, thus releasing extra A out of the membrane into the B phase. Since the total rate of A transport through the membrane consists of the rate of A diffusion and the rate of AB diffusion, the net transport of A is facilitated. It is also easy to explain the limiting rate of facilitated diffusion that does not depend on the increase of the concentration gradient A based on this model. If the chemical reaction is the rate-determining step, the overall transport rate of A will be determined by the reaction rate and the capacity of the reaction in...
the membrane. Therefore, the increase of the concentration A produces little
effect on the overall rate of A transport.

A detailed theoretical and experimental investigation of nitric oxide diffusion
through a liquid film of ferrous chloride solution was published by Ward [39].
The basic governing equations are three transport equations for A, B, and AB:

\[
D_A \frac{d^2 C_A}{dx^2} = k_1 C_A C_B - k_2 C_{AB} , \tag{3.41}
\]

\[
D_B \frac{d^2 C_B}{dx^2} = k_1 C_A C_B - k_2 C_{AB} , \tag{3.42}
\]

\[
D_{AB} \frac{d^2 C_{AB}}{dx^2} = -k_1 C_A C_B + k_2 C_{AB} . \tag{3.43}
\]

With appropriate boundary conditions, the simultaneous differential equations
are solved for these cases. His experimental data confirm the theoretical analysis.

The most recent publication dealing with carrier-facilitated transport, by
Smith et al. [40], deals with the physical and mathematical characteristics of
the process. The rather extensive analysis resulted in mathematical formulations
that permit interpretation of available data, even with wide variations in
operating parameters.

15 ACTIVE TRANSPORT

All the phenomena discussed above are for nonliving membranes. Mass
transport takes place in the direction from high concentration to low
concentration, or from high electrochemical potential to low electrochemical
potential. For living membranes, such as cell membranes, the transport
phenomena are generally quite complicated. In some cases, a substance may be
transported through a cell membrane against a concentration gradient or electrochemical potential gradient. This does not mean that the second law of thermodynamics is violated. In addition to the diffusion process, there must be other mechanisms that cause this phenomenon by supplying energy to the system. Thus, the cell is doing work to move the substance through the cell membrane against a concentration gradient. Since the cell membrane is "actively" transporting the matter, the term "active transport" is used for such a process. Some authors use the phrase "passive transport" for all the other permeation processes that are taking place in the same direction as the concentration gradient in order to contradiquish the two types of transport processes. This is shown in Fig. 3.4; the arrows indicate the directions of

```
High Concentration

Passive Transport

Membrane

Active Transport

Low Concentration
```

Fig 3.4 Diagrammatic scheme for active transport.

material transport. Since active transport requires extra energy released by metabolic reactions, it is often called "metabolically linked transport." A whole chapter is devoted to this subject in the excellent book *Cell Physiology* by Giese [41]. A theoretical treatment of the active transport based on the nonequilibrium thermodynamics will be found in a chapter of Katchalsky's book [34].

Numerous examples have been cited in the literature, and many different mechanisms have been suggested to explain the active transport. However, only a generalized (but simplified) scheme will be given here to show how the active transport is possible for cell membranes. A model scheme is depicted in Fig. 3.5, which is quite similar to Fig. 3.3 for the facilitated diffusion. But the main difference is in their directions of mass flow with respect to the concentration gradient. Another different aspect of the active transport model lies in the fact that the second component B does not have to be involved in the overall permeation processes, instead, it plays an indispensable role in the metabolic cycle of active transport within the cell membrane. Suppose that there is a certain enzyme $E_1$ near the surface of low-concentration A. This enzyme promotes the chemical reaction between A and B to yield a compound AB within the cell membrane. According to its concentration gradient, the compound AB will diffuse through the membrane to the other side, where there
is another enzyme $E_2$. The enzyme $E_2$ does just the opposite of what $E_1$ does, that is, it expedites the decomposition of AB into A and B. Then the component A will diffuse out of the membrane to the high-concentration side, because there is a buildup of A locally inside the membrane. The carrier molecule B is too large to pass through the cell membrane, thus being contained within the membrane. Therefore, these metabolic reactions together with appropriate enzymes make an engine that generates power to pump the component A against its concentration gradient.

The accumulation of potassium ions in cells is a well-known example of active transport. In many plant and animal cells, the accumulation of potassium has been found up to one thousand times higher than the ambient medium. Another example is the active transport of dyes through the cells of the kidney tubule. A carefully prepared kidney tubule is placed in a dilute solution of phenol red. After awhile, the dye is accumulated in the cavity showing deep pink in color. But placing this on ice, the dye diffuses out and makes a homogeneous color. Warming up enhances the accumulation of the dye again, which strongly suggests that the metabolic supply of energy is valid in active transport.

Nomenclature

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<td>$\bar{f}$</td>
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<td>Equilibrium concentration</td>
</tr>
</tbody>
</table>
\( e \) \hspace{1cm} \text{Porosity}
\( \xi \) \hspace{1cm} \text{Zeta potential}
\( \lambda \) \hspace{1cm} \text{Specific conductivity}
\( \mu \) \hspace{1cm} \text{Viscosity}
\( \pi \) \hspace{1cm} 3.1415
\( \pi_i \) \hspace{1cm} \text{Osmotic pressure, } i = A, B, \ldots
\( \phi \) \hspace{1cm} \text{Rate of lost work}

\textbf{Subscripts}

1,2 \hspace{1cm} \text{Position indicator; also phase designation}

\( A, B, i, j \) \hspace{1cm} \text{Components}

\( M, m \) \hspace{1cm} \text{Membrane property}

\textbf{References}