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MEMBRANE SEPARATIONS IN CHEMICAL PROCESSING

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National Bureau of Standards
U.S. Department of Commerce
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Rapidly rising energy and operating costs have underscored the need for novel energy efficient separations processes, such as membrane processes, which avoid the energy consuming phase change step of many conventional separations (e.g., distillation, absorption, stripping). It is well known that membrane separation is the most energy efficient separation technique thermodynamically possible, since it does not rely upon vaporization and condensation to effect fractionation. Recent developments in solid polymer, hollow fiber and liquid membranes give excellent promise for industrial application. Hence, because of recent technological developments of these new membranes, and because of its great energy savings potential, research is being conducted on the fundamentals determining separation rates, separation factors, and selectivity of new types of solid and liquid membranes with potential application to the chemical process industry.

Key words: chemical engineering; facilitated transport; liquid membrane; membrane; purification; separation.

1.0 Introduction

In a typical reaction process in the chemical process industry (CPI), 70% of the capital costs and 80% of the energy consumed are used in separation and concentration steps (Chem. and Eng. News, 7-28-80). Fractional distillation has been the separation technique of choice, and doubtlessly will continue to be so, because of its proven reliability and predictability. Distillation is, however, energy intensive, requiring as it does the phase changes of boiling and condensing. Therefore, it has become appropriate to examine less energy intensive separation techniques. One of these is the separation of components by membranes.

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A membrane is a thin section of material that acts as a phase separator, yet is permeable to components in solution, such as gas mixtures and liquid solutions. The membrane may be a solid, such as natural cell walls in biological processes, or the synthetic polymers used for gas separations and water desalinization (reverse osmosis). Recently (Li, 1968, 1971), attention has been focused on liquid membranes. In these systems, a liquid solution which may contain a preferential carrier is immobilized on a solid substrate (thin sponge), or stabilized by surfactants in an emulsion. Each of these two broad types, solid and liquid membranes, will be discussed below.

2.0 Solid Polymer Membranes

    Consider a multicomponent fluid mixture in contact with one surface of a membrane barrier (e.g., a solid, homogeneous film). On the other side of the membrane is another fluid mixture. If the partial molar free energies (chemical potentials) of one or more components common to both fluids differ across the membrane, then there will generally be a transfer of components through the membrane in the direction of declining chemical potential for each. If the "upstream" and "downstream" fluids are gas or vapor mixtures at differing hydrostatic pressures, for example, components pass through the barrier in the direction of declining partial pressure by a process usually termed gas permeation. If the "upstream" fluid is a liquid mixture and the "downstream" fluid is a gas in which the partial pressures of the permeating components are lower than the vapor pressures over the components in the upstream liquid, transmembrane permeation occurs by a process often termed pervaporation. If liquid phases are in contact with the membrane at equal hydrostatic pressures, components can transfer across the barrier under the action of a concentration difference between the contracting liquids by the process of dialysis. If a

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liquid mixture is confined at high hydrostatic pressure on one side of a
barrier where lower hydrostatic pressure is maintained on the other side,
certain components of the upstream liquid will permeate the membrane by the
process of ultrafiltration or reverse osmosis.

All of these membrane transport processes have the common features of:
(a) transport of mass by the action of a free-energy driving force, and (b)
the capacity to alter mixture composition by virtue of the ability of the
membrane barrier to pass one component more rapidly than another, despite
equality of driving potential. It is this latter unique characteristic of
membrane separation which differentiates this process from most common separa-
tion operations (Michaels and Bixler, 1968).

Thus the synthetic polymeric membranes can discriminate between sub-
stances on the basis of several physical properties. In the process called
ultrafiltration, molecules are separated according to their size; in elec-
trodialysis according to their electric charge; in solvent extraction accord-
ing to their differing chemical potential in solution. Membranes that are
permeable almost exclusively to water are employed in the process called
reverse osmosis. With an arrangement of several membranes, a solution of a
salt in water can be decomposed into an acid and a base, and by binding an
enzyme to a membrane, chemical reactions can be promoted with efficient
separation of reactants and products (Gregor, 1978).

2.1 Ultrafiltration

Perhaps the most straightforward membrane technology is ultrafiltration,
in which dissolved molecules and suspended particles are sorted out primarily
according to whether or not they are small enough to pass through pores in the
membrane. In ordinary filtration, a liquid is forced under pressure through a

porous medium, such as a sheet of paper or a bed of packed sand. Ultrafiltration is not fundamentally different, except that the pores are roughly 1,000 times smaller than those in an ordinary filter, so that not only particulate matter but also selected molecules can be removed and concentrated.

Ultrafiltration is in essence molecular filtration; it discriminates between molecules of different sizes, and roughly speaking between those with different molecular weights.

An ultrafiltration membrane is made, or "cast," from a solution of long-chain polymers, such as cellulose acetate dissolved in acetone. The solution is viscous (rather like honey), and it is spread with a knife edge so as to form a thin layer. Some of the solvent is then allowed to evaporate, with the result that the polymer starts to come out of solution, creating a thin, semisolid matrix. Finally the membrane is plunged into another solvent, such as water, that rapidly precipitates all of the remaining polymer. It is this quenching that forms the pores in the membrane, as the rapid precipitation leads to the clumping or coagulation of the polymer.

Loeb discovered a method of casting membranes that are both thin enough and tight enough for practical reverse osmosis (Loeb, 1966). His method takes advantage of a phenomenon observed when a membrane is cast by allowing a solvent to evaporate: after an initial period of drying, a skin appears because the solution exposed at the surface dries faster than the layers underneath. The formation of such a skin is ordinarily avoided, so as to create a membrane of uniform porosity. Loeb deliberately created a skin, then plunged the membrane into water to coagulate the polymers in the deeper layers that had not yet completely dried. The result was a membrane with an extremely thin film of tight, coherent polymer over a much thicker layer of highly porous material. Such membranes are strong enough to withstand high pressure,

but their active skin is only about 1 percent as thick as that of an ordinary ultrafiltration membrane. As a result the flux of water is 100 times greater.

2.2 Reverse-osmosis

Probably the largest single application of polymer membranes is the desalinization of water through reverse-osmosis.

In a reverse-osmosis process, the object is to make water flow selectively out of a concentrated salt solution, through a polymeric membrane, and into a solution of low salt concentration. The natural process of osmosis would cause the water to flow in the opposite direction until a pressure imbalance equal to the osmotic pressure is built up. The osmotic pressure is proportional to solute activity, and hence is approximately proportional to the salt concentration; for natural seawater the osmotic pressure is about 2.5 MPa (King, 1980). This means that in the absence of other forces, water would tend to flow from pure water into seawater--across a membrane permeable only to water--until a static head of 2.5 MPa, or 258 m, of water was built up on the seawater side. This assumes that the seawater is not significantly diluted by the water transferring into it. If the pressure difference across the membrane were less than the osmotic pressure, water would enter the seawater solution by osmosis. When the pressure difference across the membrane is greater than the osmotic pressure, water will flow from the seawater solution into the pure-water side by reverse osmosis. For the water passing through the membrane to be salt-free in a reverse-osmosis process, the membrane must be permeable to water but relatively impermeable to salt, since the salt would pass through the membrane with the water if the membrane were salt-permeable.

A number of devices have been used to evaluate different membrane materials for the desalination of seawater by reverse osmosis. It has been found (Merten, 1966) that the flux of water and salt through a membrane can be described by two equations

$$N_W = k_W(\Delta P - \Delta\pi)$$

$$N_S = k_S(C_{S_1} - C_{S_2})$$

where N_W, N_S = water and salt fluxes, respectively, across membrane, mol/time-area

ΔP = drop in total pressure across membrane

$\Delta\pi$ = drop in osmotic pressure across membrane

C_{S_1}, C_{S_2} = salt concentrations on two sides of membrane

k_W, k_S = empirical constants depending on membrane structure and nature of salt.

If the membrane has a low salt permeability $C_{S_2} \approx C_{S_1}$), we can derive the separation factor for the membrane from the above equations as:

$$\alpha_{W-S} = \frac{C_{S_1}}{C_{S_2}} \frac{C_{W_2}}{C_{W_1}} \approx \frac{N_W}{N_S} \frac{C_{S_1}}{\rho_W} = \frac{k_W(\Delta P - \Delta\pi)}{\rho_W k_S}$$

if C_{S_1} is expressed in moles per liter and ρ_W is the molar density of water in moles per liter. Since the separation factor for a given k_W and k_S should depend upon ΔP and C_{S_1} (through $\Delta\pi$), it is possible to compare the separation factors of different membranes at the same ΔP and C_{S_1}.

2.3 Capacity of Polymeric Membranes

The capacity (flux) of an ultrafiltration or reverse osmosis membrane can be measured in terms of the volume transmitted by a unit area of membrane in a given period and at a given applied pressure. As expected, the flow increases as the pores become larger. The practical unit is gallons per square foot per day at an applied pressure of 100 pounds per square inch; the metric unit is micrometers per second at a pressure difference of one 0.987 bar (1 atmosphere).

Directly measuring the size of the pores in a membrane is a difficult procedure, but the size can be estimated by measuring the rate of flow under standard conditions, which is determined largely by pore size. The measured flux can be calibrated by observing the rejection of molecules of various sizes by a membrane with a known rate of flow. Typical test molecules might be beef serum albumin (a protein of molecular weight 75,000), the dye erythrosin (molecular weight 800) and sucrose (molecular weight 342). A "tight" membrane, with a flux of one micrometer per second at one 0.987 bar (1 atmosphere) (or equivalently 14 gallons per square foot per day at 100 pounds per square inch) rejects the larger molecules almost completely and excludes some of the sugar. A membrane of average flux, approximately three micrometers per second at one atmosphere, rejects the dye and the protein but allows most of the sugar to pass. A membrane characterized as "fast," with a flux of from 10 to 20 micrometers per second, rejects only the proteins, and one that is very fast (greater than 50 micrometers per second) allows even the protein to pass but excludes particulate matter such as microorganisms.

2.4 Hollow Fiber Polymeric Membranes

Such membranes take the form of flat sheets as in leaf type filter presses, or may be spiral wound. In another configuration, the membrane is spun in the form of a hollow fiber, usually no thicker than a human hair.

Because the fibers have thick walls compared to their overall dimensions they can usually withstand pressures of as much as 10 MPa (1,500 psi). The cost is about the same as it is for the spiral-wound construction.

Such hollow fiber membrane separators have been successfully applied to separate H_2 from process or purge gas streams in the chemical process industry (Narayan, 1981). They have been demonstrated in H_2/CO_2-CO systems, H_2 recovery from ammonia plant purge gas streams, and H_2 recovery from other hydrogenation processes.

These membrane separators physically resemble a shell and tube heat exchanger. A bundle of hollow fibers is plugged at one end with the other open end being cast in a tube sheet. This bundle is housed in a steel shell with appropriate inlet and outlet piping. The feed gas containing hydrogen and other components enters through the shell feed inlet and passes along the outside surface of the hollow fibers. The hydrogen permeates through the fiber wall and is recovered from the open end of the bores. The hydrogen depleted non-permeate stream exits through the shell outlet piping. The driving force for permeation is the difference in hydrogen partial pressure between the shell and bore sides of the fiber.

Similar hollow fiber membrane processes have been developed by Canadian and Japanese companies for the recovery of helium from natural gas.

The Canadian process was developed in Edmonton (Business Week, 1974). The technology was used (Chemical Week, 1978) to build a pilot scale facility in 1978 consisting of a three-stage hollow fiber permeation train operating 5.9 to 6.9 MPa (850-1000 psi) to produce 90% helium. The pilot plant was scheduled to run until March 1981 at which time a decision would be made concerning the commercialization of the process.

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The Japanese have also developed a membrane process for helium recovery (Chem. and Eng. News, 1972). The process employs hollow fiber membranes operating at room temperature and 2.9 MPa (425) psi. It is claimed that the membrane process will reduce operating costs by 30% when compared with conventional helium recovery processes.

3.0 Liquid Membranes

An effective method for increasing the selectivity and throughput capacity of a membrane in a separation process is to incorporate a substance which reacts chemically with the component to be transmitted preferentially (Robb and Ward, 1967; Ward, 1970; Reusch and Cussler, 1973). The chemical reaction increases the concentration of the component within the membrane, and thereby increases the concentration-difference driving force for diffusion of the component through the membrane. Such systems are referred to as Facilitated Transport Membranes, and the current activity in this field is largely due originally to Norman Li (Li, 1966).

The liquid membrane technique is simultaneous extraction/stripping process by means of a selective liquid membrane separating two other miscible liquid phases.

The term liquid membrane itself is used to denote that component of, in general, three liquid phases which separates spatially an external phase present in excess (either continuous feed or batch process) from a lean phase. Transport of a dissolved material through the membrane phase can take place from the external phase into the lean phase, or in the opposite direction. This process is termed permeation. In general, membrane processes are characterized by the driving force and the type of transport process which are present. Thus, as discussed above, filtration, ultrafiltration, and reverse

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osmosis represent convective mass transport processes, the driving force for which is a pressure gradient across the membrane.

In contrast to these processes, the mass transport selectivity and transfer rate of a liquid membrane are determined primarily by the solubilities and diffusion coefficients of the permeant in the membrane phase. The driving force of the transport process is the chemical potential gradient, which is the concentration gradient in the simplest case.

Liquid membranes may take one of two forms (Fig. 1). In the first, the liquid membrane containing a carrier chemical is impregnated on a polymer support which acts like a sponge. Such systems are called Immobilized Liquid Membranes (ILM's) (Fig. 1-a). In the second form, the liquid membrane is stabilized with surfactants and stirred into the lean phase. An emulsion results, in which the liquid membrane surrounds and encapsulates the lean phase, like the shell of a jelly bean. Such systems are called Emulsion Liquid Membranes (ELM's) (Fig. 1-b).

3.1 Immobilized Liquid Membranes

The diffusion coefficient of most molecules in polymers is so low that exceedingly thin polymeric membranes must be constructed to produce industrially acceptable fluxes. These thin membranes may have fabrication and mechanical integrity problems. Selectivity has usually been poor in membrane systems unless the mixture components are radically different, as in desalination. On the other hand, diffusion coefficients in liquid films are sufficiently large that fluxes become industrially interesting. Specific chemical carriers can be incorporated into these immobilized liquid films to give the desired selectivity toward one component of a mixture. Work is now being directed toward applying this new class of membranes to large scale operations.

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Immobilized facilitated transport membranes hold excellent promise for industrial application. Indeed, early studies show potential cost savings of 30% to 50% over common acid scrubbing of CO_2, and energy savings as much as a factor of 3 (Kimura et al., 1979). For instance, conventional processes for scrubbing CO_2 require absorption at feed stream pressure and subsequent regeneration at reduced pressure (Fig. 2). Such processes may involve high solution circulation rates and large pumping power requirements, and thus are capital intensive. A schematic diagram of an equivalent CO_2 immobilized liquid membrane scrubber is shown in Figure 3-a. The facilitated transport membrane can be viewed as an absorber on the high-pressure side and a stripper on the low pressure surface. Thus, the membrane system is analogous to a conventional scrubbing system, but requires only a single vessel. Also, need for circulation of a large volume of absorbent liquid is eliminated.

A schematic representation of the CO_2 facilitated transport occurring in the liquid membrane is shown in Figure 3-b.

The important reactions occurring are:

$$CO_2 + H_2O \rightarrow H^+ + HCO_3^-$$

$$CO_3^= + H^+ \rightarrow HCO_3^-$$
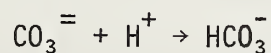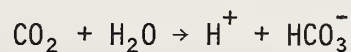
Addition of these two reactions yields the net results:

$$CO_2 + CO_3^= + H_2O \rightarrow 2HCO_3^-$$
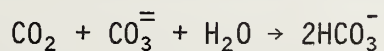
This reaction occurs on the high-pressure side of the membrane while the reverse reaction occurs when CO_2 is rejected on the low-pressure side of the membrane regenerating the carbonate complex.

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In practice, a porous support (typically cellulose acetate) could be impregnated with a nonvolatile liquid membrane such as a carbonate solution. Reactants and/or catalysts in the membrane react with the solute and carry it to the solute lean side of the membrane, where the reaction is reversed and the solute is expelled. In this way the selectivity for solutes is markedly increased, in the case of CO_2, by as much as by a factor of 10^3.

Recently, Hughes et al. (1981) presented results of a pilot scale study of ethylene and propylene transport using immobilized liquid membranes. Cellulose acetate hollow fibers were impregnated with an aqueous solution of silver nitrate. The silver ions acted as the complexing agent in the facilitated transport process. The pilot plant was based on a commercial size (18.6 m^2 membrane area) hollow fiber permeation vessel and was fed a vent stream from a polypropylene plant containing 75% propylene. The pilot plant succeeded in producing a 98% purity propylene gas product. Hughes et al. reported no current plans to commercialize this process further.

3.2 Emulsion Liquid Membranes

The other form of facilitated transport liquid membranes is the ELM, shown schematically in Figure 4.

The upper part, (a), of Figure 4 shows an idealized model representation of a liquid membrane capsule. In the lower part, (b), a realistic liquid membrane is sketched. The membrane itself separates two miscible phases from each other, and in insoluble in these phases. It can have a hydrophilic or a lipophilic character. In the case sketched here, such as for the removal of phenol from a continuous aqueous phase, the membrane can consist of a hydrocarbon mixture. It is stabilized by a surfactant with a suitable value of the

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hydorphilic-lipophilic balance (HLB). The typical diameter of a liquid-
membrane globule lies in the order of magnitude range for 10 μm to 1 mm.

Transport of the desired component across the ELM is the same as above,
the driving force being the difference in chemical potential of the component
between the outer and inner phases. On the inside of the liquid membrane
capsule, an attempt is usually made to modify the permeant chemically in some
way, as far as possible, in order to set up a maximum concentration gradient
across the membrane, and hence to maximize the driving force of the transport
process. The optimum volumetric ratio of the dispersed phase to the membrane
phase is specific to the system (1:1 to 10:1). Compared with traditional
extraction techniques, up to 90% of the cost of the solvent phase can be
saved. The introduction of liquid-membrane techniques can therefore also be
economically interesting even in those cases where relatively expensive
membrane liquids such as silicone oils are needed. The membrane viscosity and
surfactant concentration are of decisive importance as regards the membrane
thickness, transfer rates, stability, and selectivity of the liquid membranes.
Figure 5 is a flowsheet for a hypothethical industrial separation process
employing the emulsion liquid membrane technique (Halwachs, 1980).

The heart of the plant is the mixer, which is fed with contaminated
water. For the removal of weak acids, such as phenol, hydrogen sulfide,
hydrocyanic acid, acetic acid, mercaptans, or other weak organic acids,
caustic soda solution should be introduced as the disperse phase. For the
removal of weak bases, such as ammonia or amines, dilute sulfuric acid should
be considered for encapsulation.

Kitagawa et al. (1976) have reported the use of licensed EML technology
to remove toxic ionic species such as Cr^{+6}, Hg^{2+}, Cd^{2+} and Cu^{2+} from indus-
trial wastewater. Pilot plant studies to optimize the ELM process for indus-
trial wastewater treatment continued into 1979 (Hayworth, 1981).

After the actual separation of the materials, there is still the treat-
ment of the multi-emulsion to be considered. The latter is first passed to a
settler unit, in which the liquid membrane phase and the continuous aqueous
phase are separated on the basis of the density difference. The aqueous phase
is then in the form of a purified wastewater, which may be subjected to
further after-purification processes as required. The recovered emulsion
phase must be broken, in turn, and the disperse phase recuperated or dis-
carded. The membrane phase, as the most valuable component of the multi-
emulsion, is passed into an emulsifier, mixed with fresh phase to be encapsu-
lated, and introduced again into the mixer.

Analogous processes serve for the oxidation of ethylene to vinyl acetate
by means of an acetic acid--palladium chloride--copper chloride membrane
phase, for the oxidation of propylene with oxygen to acetone, for the oxida-
tion of acetaldehyde with oxygen to acetic acid, for the carbonylation of
methanol with carbon monoxide to acetic acid, and for hydroformylation of
propylene with carbon monoxide and hydrogen to n-butyraldehyde (Halwachs,
1980).

Another promising application, where, indeed, the center of gravity of
possible ELM applications may lie, is in metallurgy. ELM's may advantageously
be used for the recovery of copper and uranium for dilute ores, and for the
concentration of strategic metals. It has been estimated (Cahn and Li, 1980)
that ELMS could reduce the capital and operating costs of copper recovery by
up to 40% when compared with solvent extraction.

4.0 Conclusion

Rapidly rising energy and operating costs have underscored the need for
novel energy efficient separation processes, such as membrane processes, which

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avoid the energy consuming phase change step of many conventional separations (e.g., distillation, absorption, stripping). It is well known that membrane separation is the most energy efficient separation technique thermodynamically possible, since it does not rely upon vaporization and condensation to effect fractionation. Approaching this minimum energy in practice, however, is difficult. Despite considerable efforts, membrane devices have not made substantial inroads in industrial separations to date due to problems of (a) speed and selectivity, (b) predictability, and (c) reliability. Recent development in solid polymer, hollow fiber and liquid membranes give excellent promise for industrial application. Hence, because of recent technological developments of these new membranes, and because of its great energy savings potential, research is being conducted on the fundamentals determining separation rates, separation factors, and selectivity of new types of solid and liquid membranes with potential application to the chemical process industry.

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(a) **IMMOBILIZED LIQUID MEMBRANES**

| RICH PHASE | MEMBRANE + IMMOBILIZED LIQUID REAGENT | LEAN PHASE |

(b) **EMULSION LIQUID MEMBRANES**

LIQUID MEMBRANE

RICH PHASE

LEAN PHASE
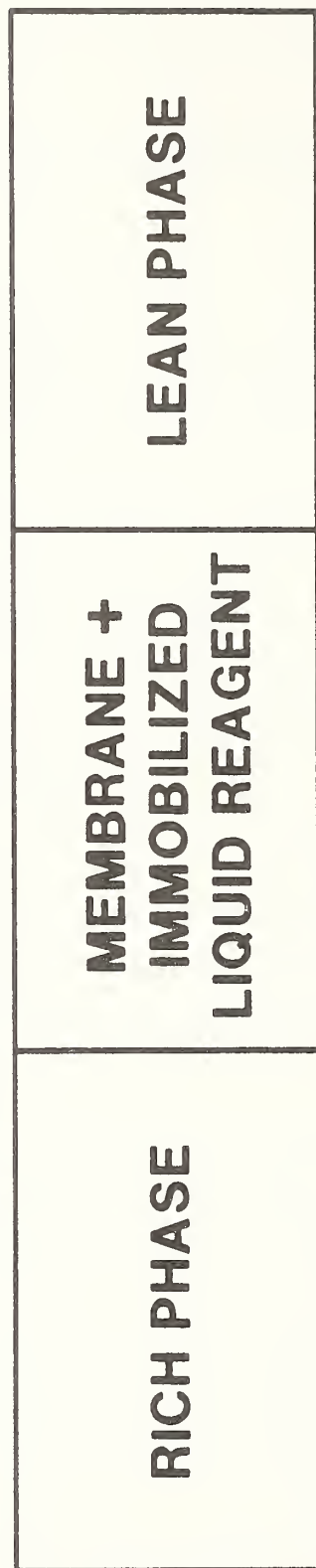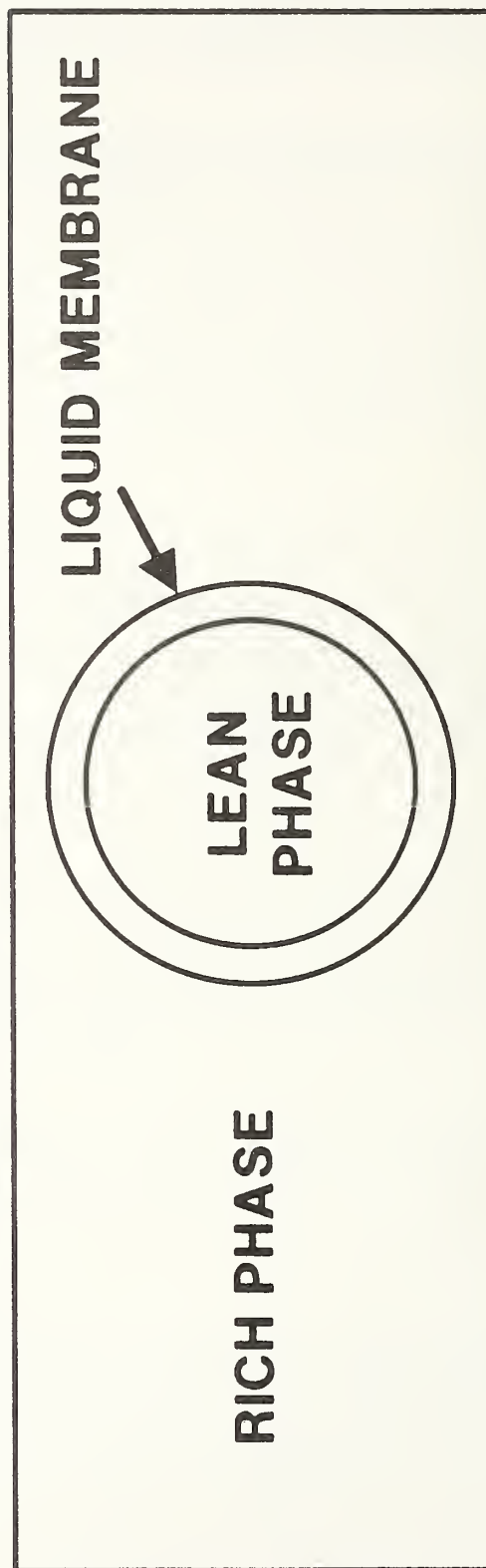
Figure 1. Facilitated Transport Liquid Membranes.

Figure 2. Conventional Acid Gas Scrubbing System
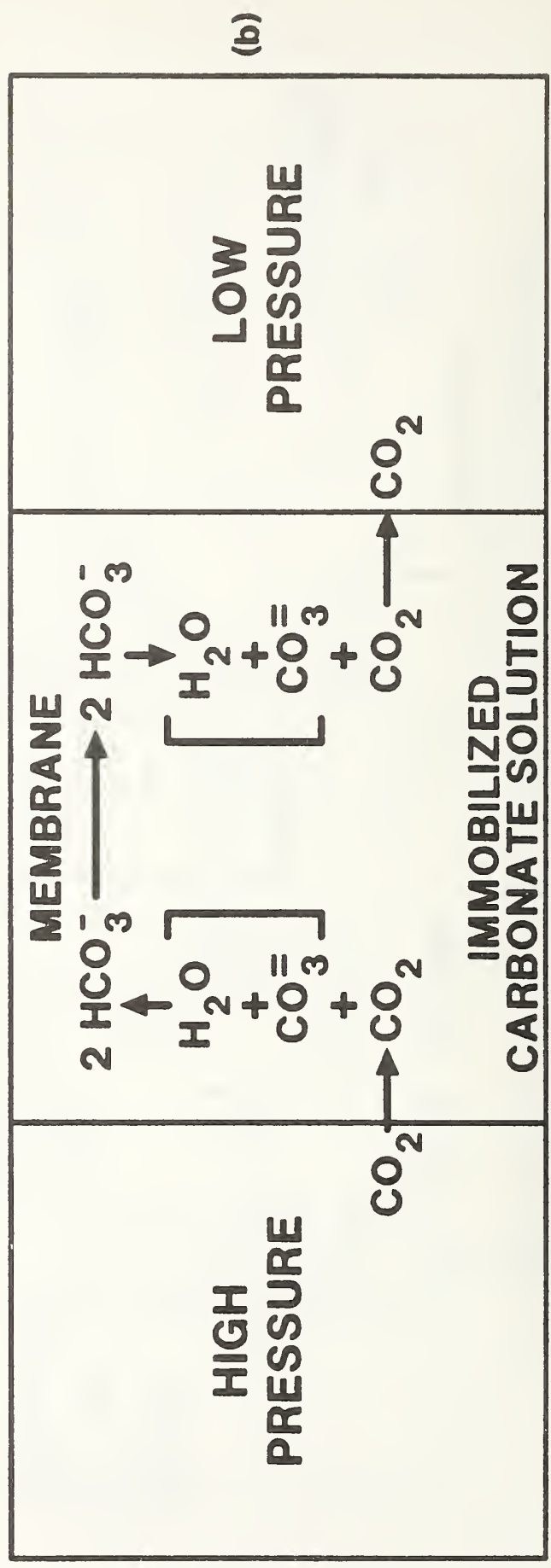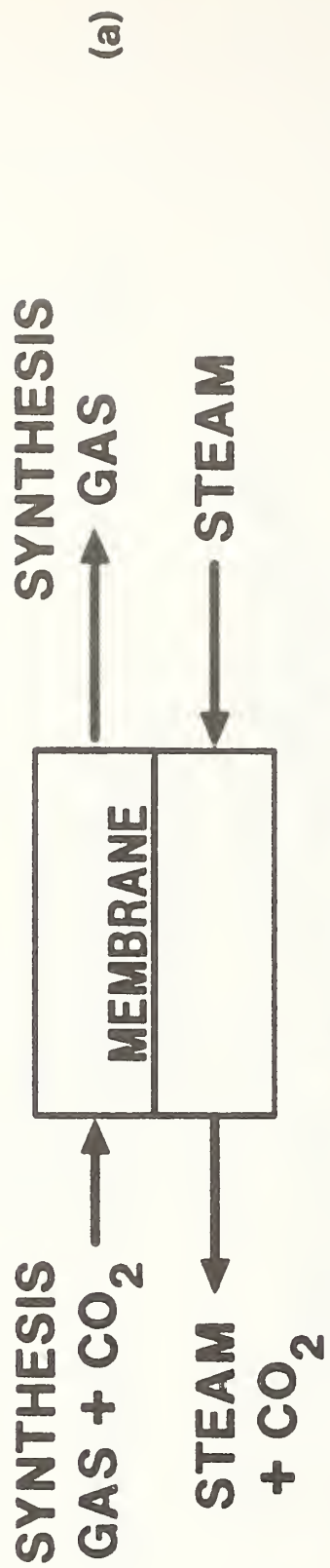
Figure 3. Membrane Carbon Dioxide Scrubber

(a) Ideal Representation

Liquid Membrane,
e.g., liquid hydrocarbon
stabilized with surfactants

Inner
Phase

Outer Phase

(b) Actual Case

Continuous Outer Phase

Inner Phase

Emulsion
Globules

0.1–5 mm
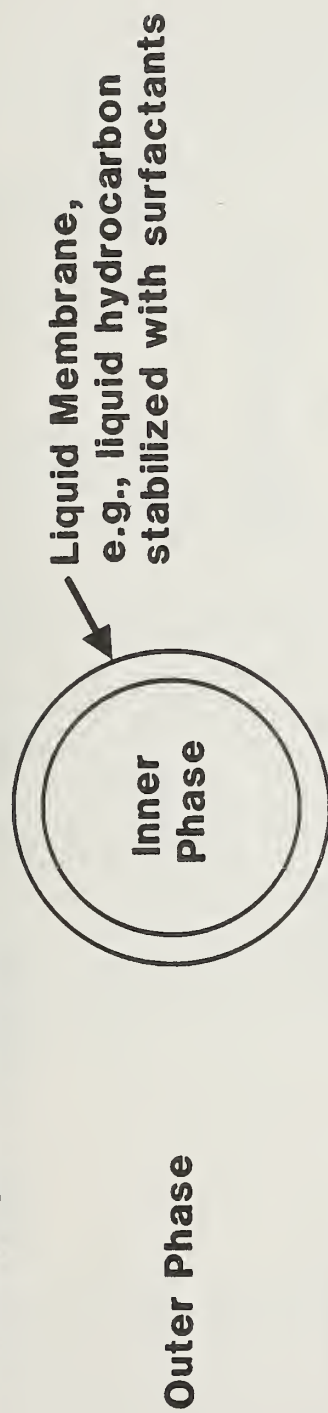
Liquid
Hydrocarbon
Surfactant
Membrane
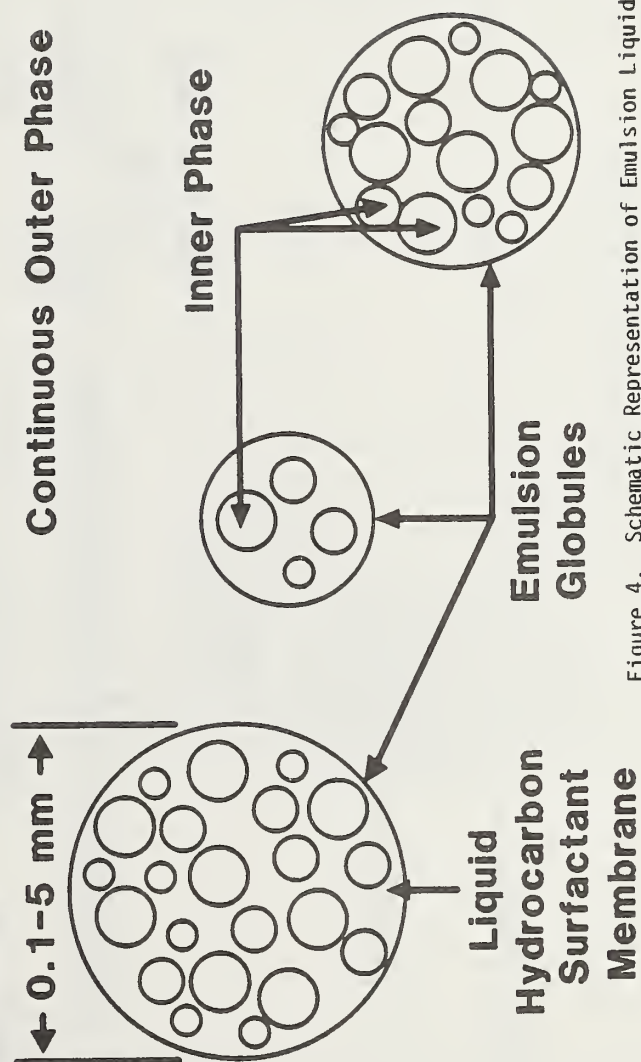
Figure 4. Schematic Representation of Emulsion Liquid Membrane (ELM)

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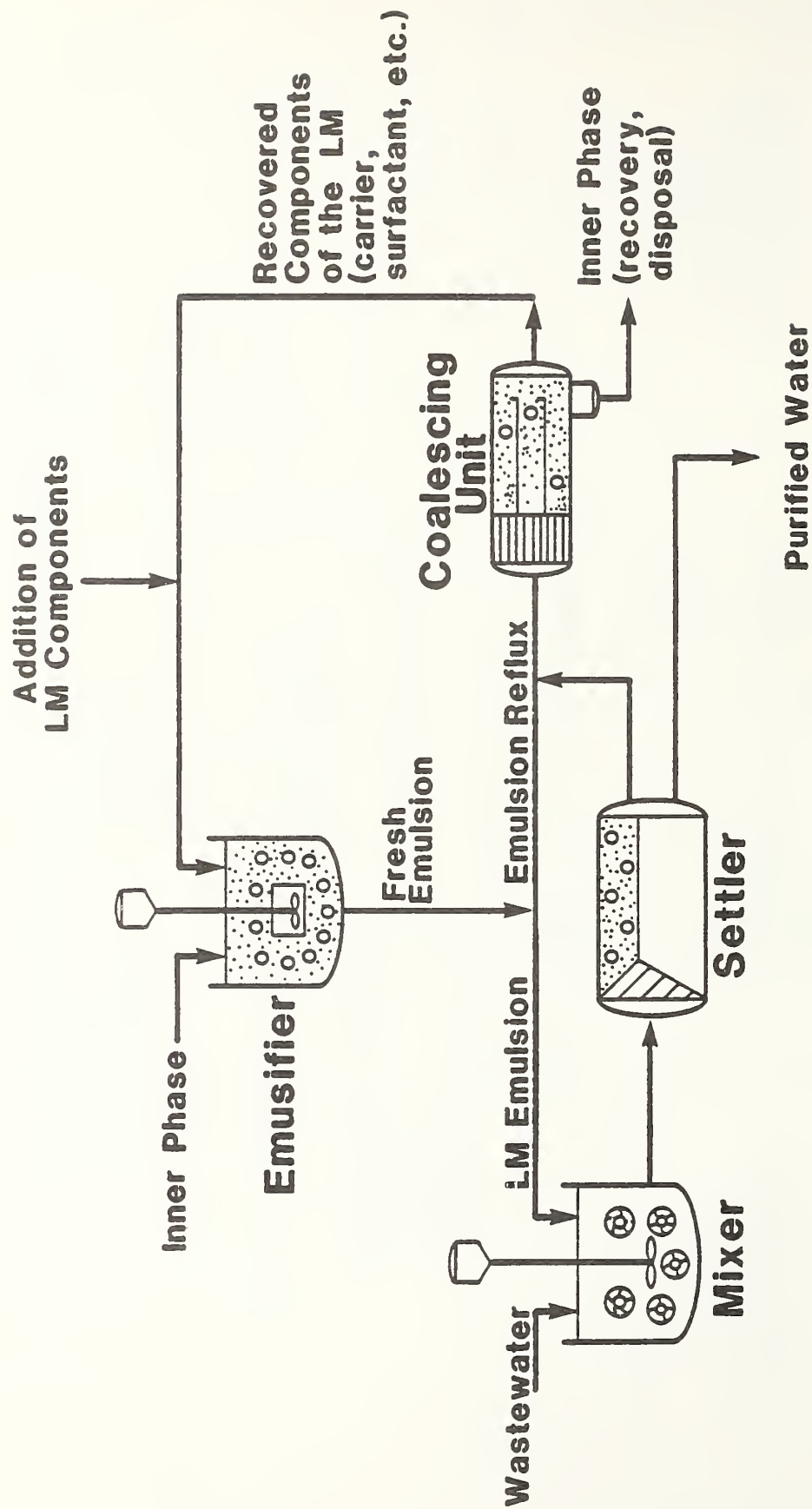
Figure 5. Representative ELM Flowsheet for Wastewater Purification

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Thomas M. Flynn and J. Douglas Way

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DEPARTMENT OF COMMERCE
WASHINGTON, D.C. 20234

7. Contract/Grant No.

8. Type of Report & Period Covered

9. SPONSORING ORGANIZATION NAME AND COMPLETE ADDRESS (Street, City, State, ZIP)

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11. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here)

Rapidly rising energy and operating costs have underscored the need for novel energy efficient separations processes, such as membrane processes, which avoid the energy consuming phase change step of many conventional separations (e.g., distillation, absorption, stripping). It is well known that membrane separation is the most energy efficient separation technique thermodynamically possible, since it does not rely upon vaporization and condensation to effect fractionation. Recent developments in solid polymer, hollow fiber and liquid membranes give excellent promise for industrial application. Hence, because of recent technological developments of these new membranes, and because of its great energy savings potential, research is being conducted on the fundamentals determining separation rates, separation factors, and selectivity of new types of solid and liquid membranes with potential application to the chemical process industry.

12. KEY WORDS (Six to twelve entries; alphabetical order; capitalize only proper names; and separate key words by semicolons)

chemical engineering; facilitated transport; liquid membrane; membrane; purification; separation.