

MEMBRANE SEPARATION SYSTEMS

A Research Needs Assessment Executive Summary

April 1990

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MEMBRANE SEPARATION SYSTEMS

- A RESEARCH & DEVELOPMENT NEEDS ASSESSMENT

by the

Department of Energy Membrane Separation Systems

Research Needs Assessment Group

Volume I

March 1990

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PREFACE

The Department of Energy has maintained a long standing interest in any energy savings that can be obtained by utilizing membrane separations systems. National interest in these systems has been mushrooming. The Department funds approximately \$4.5 million in membrane research and development in five different programs ranging from the Office of Industrial Programs in the Office of Conservation Energy Programs to the Office of Energy Research in the Division of Chemical Sciences.

In all these efforts, the Department seeks to reduce the technological barriers to widespread acceptance to industrial utilization of membrane based separation technology replacing the conventional energy-intensive "workhorse" processes of distillation, evaporation, filtration and sedimentation. Before new applications can reach commercial usability, additional research is required.

The study offers the membrane research community a blueprint, for the next 5 to 20 years, to further the applications of membrane separations for greater potential energy savings. According to a recent DOE study (DOE/NBM-80027730), energy savings of over 1 quad, equivalent to 170 million barrels of oil annually, could be achieved if membrane separations were utilized in the liquid-to-vapor separations alone, out of a total of 2.6 quads expended.

This report outlines, in considerable detail, the 38 highest priority research needs in seven major categories including reverse osmosis, ultrafiltration, microfiltration, electrodialysis, pervaporation, gas separation and facilitated transport. International activities are also covered.

Separation processes utilizing membrane technology, are relatively new and rapidly being utilized in industrial processes for energy savings applications ranging from the food processing industry to the chemical industry. It shows real potential.

Careful understanding of the research barriers to be overcome can translate into significant energy savings and improved national productivity. We commend Dr. William Eykamp and his colleagues for their suggestions for setting the Nation's direction in membrane research.

ABSTRACT

Membrane based separation technology, a relative newcomer on the separations scene, has demonstrated the potential of saving enormous amounts of energy in the processing industries if substituted for conventional separation systems. Over 1 quad annually, out of 2.6, can possibly be saved in liquid-to-gas separations, alone, if membrane separation systems gain wider acceptance, according to a recent DOE/OIP (DOE/NBM-80027730 (1986)) study. In recent years great strides have been made in the field and offer even greater energy savings in the future when substituted for other conventional separation techniques such as distillation, evaporation, filtration, sedimentation, and absorption.

Consequently, the Office of Program Analysis (OPA) of the Department of Energy's (DOE's) Office of Energy Research, sponsored an assessment of the research still needed to bring energy-saving membrane separation processes to technical and commercial readiness for commercial acceptance within the next 5 to 20 years. This assessment was conducted by a group of six internationally known membrane separations experts who examined the worldwide status of research in the seven major membrane areas. This encompassed four mature technology areas: reverse osmosis, microfiltration, ultrafiltration, and electrodialysis; two developing areas: gas separation and pervaporation; and one emerging technology: facilitated transport.

Particular attention was paid to identifying the innovative processes currently emerging, and even further improvements which could gain wider acceptance for the more mature membrane technology. In all, 38 priority research areas were selected and ranked in order of priority, according to their relevance, likelihood of success, and overall impact. Rationale was presented for all the final selections. This study was peer reviewed by an additional ten experts. This study reviews the finding and research recommendations developed in this assessment, based upon a worldwide analysis of membrane separations science and an assessment of current U.S. and DOE membrane activities.

The topics that were pointed out as having the greatest research emphasis are pervaporation for organic-organic separations; gas separations; microfiltration; an oxidant-resistant reverse osmosis membrane; and a fouling-resistant ultrafiltration membrane.

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CHAPTER ONE

EXECUTIVE SUMMARY

The Office of Program Analysis in the Office of Energy Research of the Department of Energy (DOE) commissioned this study to evaluate and prioritize research needs in the membrane separation industry.

One of the primary goals of the U.S. Department of Energy is to foster and support the development of energy-efficient new technologies. In 1987, the total energy consumption of all sectors of the U.S. economy was 76.8 quads, of which approximately 29.5 quads, or 38%, was used by the industrial sector, at a cost of \$100 billion.¹ Reductions in energy consumption are of strategic importance, because they reduce U.S. dependence on foreign energy supplies. Improving the energy efficiency of production technology can lead to increased productivity and enhanced competitiveness of U.S. products in world markets. Processes that use energy inefficiently are also significant sources of environmental pollution.

The rationale for seeking innovative, energy-saving technologies is, therefore, very clear. One such technology is membrane separation, which offers significant reductions in energy consumption in comparison with thermal separation techniques. Membranes separate mixtures into components by discriminating on the basis of a physical or chemical attribute, such as molecular size, charge or solubility. They can pass water while retaining salts, the basis of producing potable water from the sea. They are used for passing solutions, while retaining bacteria, the basis for cold sterilization. They can separate air into oxygen and nitrogen. There are numerous applications for membranes in the world today. Total sales of industrial membrane separation systems worldwide are greater than \$1 billion annually.² The United States is a dominant supplier of these systems. United States dominance of the industry is being challenged, however, by Japanese and, to a lesser extent, European competitors.

Some membranes are used in circumstances where energy saving is an important criterion. Others are used in small-scale applications where energy costs are relatively unimportant. This report looks at the major membrane processes to assess their status and potential, particularly with regard to energy

saving. Related technologies, for example the membrane catalytic reactor, although outside the scope of this study, are believed to have additional potential for energy savings.

This report was prepared by a group of six membrane experts representing the various fields of membrane technology. Based on group meetings and review discussions, a list of five to seven priority research topics was prepared by the group for each of the seven major membrane technology areas: reverse osmosis, ultrafiltration, microfiltration, electrodialysis, pervaporation, gas separation and facilitated transport. These items were incorporated into a master list, totaling 38 research topics, which were then ranked in order of priority.

The highest ranked research topic was pervaporation membranes for organic-organic separations. Another pervaporation-related topic concerning the development of organic-solvent-resistant modules ranked seventh. The very high ranking of these two pervaporation research topics reflects the promise of this rapidly developing technology. Distillation is an energy-intensive operation and consumes 28% of the energy used in all U.S. chemical plants and petroleum refineries.³ The total annual distillation energy consumption is approximately 2 quads.⁴ Replacement or augmentation of distillation by pervaporation could substantially reduce this energy usage. If even 10% of this energy could be saved by using membranes, for example in hybrid distillation/pervaporation systems, this would represent an energy savings of 0.2 quad, or 10^5 barrels of oil per day.

Three topics relating to the development of gas-separation membranes ranked in the top 10 of the master list. Membrane-based gas separation is an area in which the United States was a world leader. The dominant position of U.S. suppliers, and U.S. research, is under threat of erosion because of the increased attention being devoted to the subject by Japanese and European companies, governments and institutions. Increased emphasis on membrane-based gas-separation research and development would increase the probability that the new generation technology for high-performance, ultrathin membranes will be controlled by the United States. The attendant benefits would be that membrane-based gas separation would become competitive with conventional, energy-intensive separation technologies over a much broader spectrum. The energy savings that

might be achieved by membrane-based gas-separation technology are exemplified by two potential applications. If high-grade oxygen-enriched streams were available at low cost, as a result of the development of better oxygen-selective membranes, then combustion processes through industry could be made more energy efficient. Various estimates have placed the energy savings from use of high-grade oxygen enriched air at between 0.06 and 0.36 quads per year.⁵ It is estimated that using membranes to upgrade sour natural gas will result in an energy savings of 0.01 quads per year.

The second highest priority topic in the master list was the development of oxidation-resistant reverse osmosis membranes. The current generation of reverse-osmosis membranes have adequate salt rejection and water flux. However, they are susceptible to degradation by sterilizing oxidants. High-performance, oxidation-resistant membranes could displace existing cellulose acetate membranes and open up new applications of reverse osmosis, particularly in food processing. The energy use for evaporation in the food industry has been estimated at about 0.09 quads.⁶ Reverse osmosis typically requires an energy input of 20-40 Btu/lb of water removed.⁷ Assuming an average energy consumption for conventional evaporation processes of 600 Btu/lb, the substitution of reverse osmosis for evaporation could result in a potential energy savings of 0.04-0.05 quads.

In general, facilitated-transport related topics scored low in the master priority list, reflecting the disenchantment of the expert group with a technology with which membrane scientists have been struggling for the last 20 years without reaching the point of practical viability. The development of facilitated-transport, oxygen-selective, solid-carrier membranes was, however, given a high research priority ranking of four. If stable, solid facilitated-transport membranes could really be developed, they might offer much higher selectivities than polymer membranes, and have a major effect on the oxygen and nitrogen production industries.

The principal problem in ultrafiltration technology is membrane fouling. The development of fouling-resistant ultrafiltration membranes was given a research priority ranking of six. The development of fouling-resistant ultrafiltration

membranes would have a major impact on cost and energy savings in the milk and cheese production industries, for example.

Two high-priority topics cover research opportunities in the microfiltration area, namely, development of low-cost microfiltration modules and development of high-temperature solvent resistant membranes and modules. Microfiltration is a well developed and commercially successful industry, whose industrial focus has been in the pharmaceutical and food industries. Drinking water and sewage treatment are new, but non-glamorous applications for microfiltration, requiring membranes and equipment whose design concept and execution may be incompatible with the mission of the private industry participants. The potential for societal impact in this area is great, but existing microfiltration firms may not find the opportunity appealing, because of technical risks, regulatory constraints or competition from conventional alternatives.

Reverse osmosis, ultrafiltration and microfiltration are all technologies with significant energy-savings potential across a broad spectrum of industry. For example, a significant fraction of the wastewater streams from the food, chemical and petroleum processing industries are discharged as hot streams and the energy lost is estimated at 1 - 2 quads annually.⁸ The development of low-cost, chemically resistant MF/UF/RO membrane systems that could recover the hot wastewater and recycle it to the process would result in considerable energy savings. If only 25% of the energy present in the wastewater were recovered, this would result in an energy savings of 0.25 to 0.50 quads.⁹

Many of the top 10 ranked priority research topics spotlighted technology and engineering problems. In the view of the authors of the report, it appears that emerging membrane separations technologies have reached a level of maturity where progress toward competitive, energy-efficient industrial systems will be most effectively expedited by increasing DOE support of engineering or technology-based research programs. Applications-related research was viewed as equally worthy of support as fundamental scientific studies. This view was not shared unanimously by the reviewers, however. Two reviewers objected that the list of research priorities was too much skewed toward practical applications and gave a low priority to the science of membranes, from whence the long-term

innovations in membrane technology will come. One reviewer, on the other hand, felt strongly that there was too much emphasis on basic research issues, and that most of the top priority items identified in the report did not adequately address engineering issues.

During the course of the study, government support of membrane-related research in Japan and Europe was investigated. The Japanese government and the European governments each spend close to \$20 million annually on membrane-related topics. Federal support for membrane-related research and development through all agencies is currently about \$10-11 million per year. The United States is, therefore, in third place in terms of government assistance to membrane research. There was concern among some members of the group that this level of spending will ultimately result in loss of world market share.

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CHAPTER TWO
ASSESSMENT METHODOLOGY

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CHAPTER TWO

ASSESSMENT METHODOLOGY

Industrial separation processes consume a significant portion of the energy used in the United States. A 1986 survey by the Office of Industrial Programs estimated that about 2.6 quads of energy are expended annually on liquid-to-vapor separations alone.¹ This survey also concluded that over 1.0 quad of energy could be saved if the industry adopted membrane separation systems more widely.

Membrane separation systems offer significant advantages over existing separation processes. In addition to consuming less energy than conventional processes, membrane systems are compact and modular, enabling easy retrofit of existing applications. This study was commissioned by the Department of Energy, Office of Program Analysis, to identify and prioritize membrane research needs in order of their impact on the DOE's mission, such that support of membrane research may produce the most effective results over the next 20 years.

2.1 AUTHORS

This report was prepared by a group of senior researchers well versed in membrane science and technology. The executive group consisted of Dr. Richard W. Baker (Membrane Technology & Research, Inc.), Dr. William Eykamp (University of California at Berkeley) and Mr. Robert L. Riley (Separation Systems Technology, Inc.), who were responsible for the direction and coordination of the program. Dr. Eykamp also served as Principal Investigator for the program.

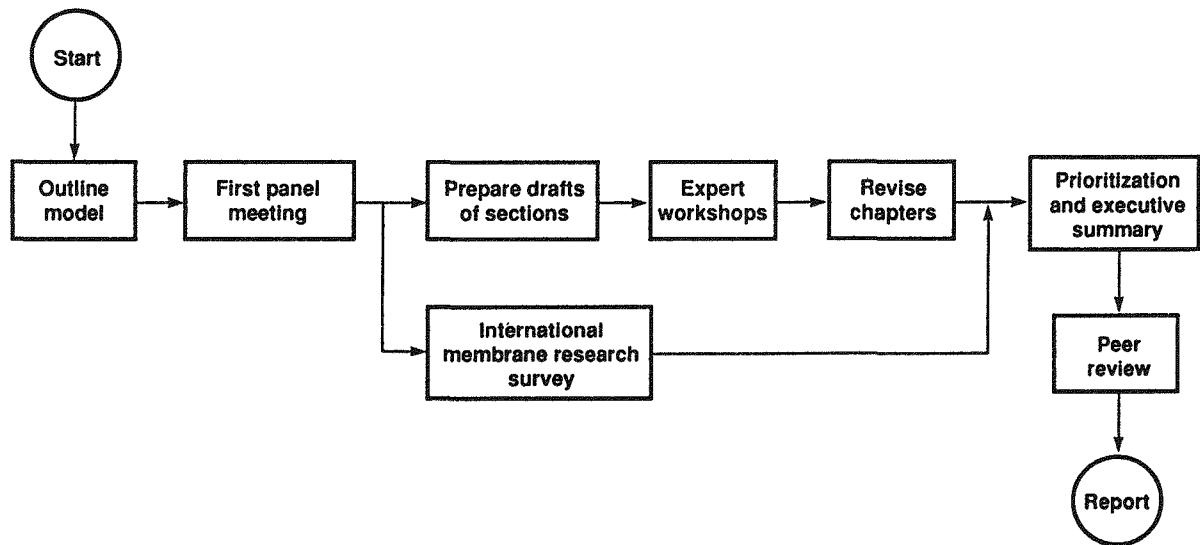
The field of membrane science was divided into seven general categories based on the type of membrane process. To ensure that each of these categories was covered by a leading expert in the field, the executive group was supplemented by three additional authors. These additional group members were Dr. Edward Cussler (University of Minnesota), Dr. William J. Koros (University of Texas at Austin), and Dr. Heiner Strathmann (Fraunhofer Institute, West Germany). Each of the authors was assigned primary responsibility for a topic area as shown in Table 2-1.

Table 2-1. List of Authors

<u>Topic</u>	<u>Author</u>
Membrane and Module Preparation	Richard Baker
Microfiltration and Ultrafiltration	William Eykamp
Reverse Osmosis	Robert Riley
Pervaporation	Richard Baker
Gas Separation	William Koros
Facilitated and Coupled Transport	Edward Cussler
Electrodialysis	Heiner Strathmann

The role of the group of authors was to assess the current state of membranes in their particular section, identify present and future applications where membrane separations could result in significant energy savings and suggest research directions and specific research needs required to achieve these energy savings within a 5-20 year time frame. The collected group of authors also performed the prioritization of the overall research needs.

As program coordinator, Dr. Amulya Athayde provided liaison between the authors and the contractor, Membrane Technology & Research, Inc (MTR). Ms. Janet Farrant (MTR) was responsible for the patent information searches and the editing and final assembly of this report. The overall plan for preparation of the report is shown in Figure 2-1.



MTR-007-WD

Figure 2-1. Overall plan for conducting the study of research needs in membrane separation systems

2.2 OUTLINE AND MODEL CHAPTER

The first major task of this program was to develop an outline for the report and draft a model chapter. The outline was prepared by the executive group and submitted to the authors of the individual sections for consideration. A patent and literature survey was conducted at MTR in each of the topic areas (listed in Table 2-1) to assess the state of the art as represented by recent patents, product brochures and journal articles. This information was provided to the group of authors.

Projects accomplished by committees are proverbially characterized by poor cohesion and a lack of direction. To circumvent such criticism of this report the section on reverse osmosis was selected as a model chapter for the rest of the report. A draft prepared by Mr. Robert Riley was circulated among the other authors to illustrate the desired format. The goal of this exercise was to ensure that the report had a uniform style and emphasis, with the individual chapters in accord with each other.

2.3 FIRST GROUP MEETING

The first group meeting was held at MTR on December 26-27, 1988, and was attended by the authors and the ex-officio group members representing the DOE: Mr. Robert Rader and Dr. Gilbert Jackson (Office of Program Analysis), Dr. William Sonnett (Office of Industrial Programs) and Dr. Richard Gordon (Office of Energy Research, Division of Chemical Sciences).

The authors presented draft outlines of their sections, which were reviewed by the entire group. The model chapter was discussed and revisions for the outlines of the other chapters were drawn up.

2.4 EXPERT WORKSHOPS

A series of "expert workshops" was held upon completion of the draft chapters to discuss the conclusions and recommendations of the authors with membrane energumena drawn from the U.S. and international membrane

communities. These workshops consisted of closed-panel discussions, organized in conjunction with major membrane research conferences.

Two or three experts in the particular area were invited to review the draft chapters and respond with their comments and criticism. The workshops provided an opportunity for the authors to update the information on the state of the art, as well as to obtain an informed consensus on the recommended research directions and needs.

The workshops for the Reverse Osmosis, Ultrafiltration, Microfiltration, Coupled and Facilitated Transport, Gas Separation and Pervaporation sections were held on May 16-20, 1989, during the North American Membrane Society Third Annual Meeting in Austin, Texas. The workshop on Electrodialysis was held on August 4, 1989, during the Gordon Research Conference on membrane separations in Plymouth, New Hampshire. A special workshop was also held at the Gordon Research Conference during which all of the authors were present and the list of research needs was discussed with the conference attendees. The lists of workshop attendees are given in Table 2-2.

Table 2-2. Workshop Attendees

WORKSHOP ON ULTRAFILTRATION AND MICROFILTRATION

Attendee	Affiliation
W. Eykamp (Author)	University of California, Berkeley
G. Jackson	DOE
R. Rader	DOE
J. Short	Koch Membrane Systems, Inc.
G. Jonsson	Technical University of Denmark
A. L. Athayde	MTR, Inc.

WORKSHOP ON REVERSE OSMOSIS

Attendee	Affiliation
R. L. Riley (Author)	Separation Systems Technology, Inc.
W. Eykamp	University of California, Berkeley
R. Rader	DOE
D. Blanchfield	DOE Idaho Operations Office
D. Cummings	EG&G Idaho
R. Peterson	Filmtec Corp.
H. F. Ridgway	Orange County Water District
A. L. Athayde	MTR, Inc.

WORKSHOP ON GAS SEPARATION

Attendee	Affiliation
W. J. Koros (Author)	University of Texas, Austin
W. Eykamp	University of California, Berkeley
R. W. Baker	MTR, Inc.
R. Rader	DOE
D. Blanchfield	DOE Idaho Operations Office
D. Cummings	EG&G Idaho
R. Goldsmith	CeraMem Corp.
B. Bikson	Innovative Membrane Systems/ Union Carbide Corp.
G. P. Pez	Air Products & Chemicals, Inc.
A. L. Athayde	MTR, Inc.

WORKSHOP ON COUPLED AND FACILITATED TRANSPORT

Attendee	Affiliation
E. L. Cussler (Author)	University of Minnesota
W. Eykamp	University of California, Berkeley
R. W. Baker	MTR, Inc.
R. Rader	DOE
G. Jackson	DOE
D. Blanchfield	DOE Idaho Operations Office
D. Haefner	EG&G Idaho
J. D. Way	SRI International
K. K. Sirkar	Stevens Institute of Technology
G. P. Pez	Air Products & Chemicals, Inc.
A. L. Athayde	MTR, Inc.

WORKSHOP ON PERVAPORATION

Attendee	Affiliation
R. W. Baker (Author)	MTR, Inc.
W. Eykamp	University of California, Berkeley
K.-V. Peinemann	GKSS, West Germany
R. Rader	DOE
G. Jackson	DOE
H. L. Fleming	GFT, Inc.
A. L. Athayde	MTR, Inc.

WORKSHOP ON ELECTRODIALYSIS

Attendee	Affiliation
H. Strathmann (Author)	Fraunhofer Institute, West Germany
W. Eykamp	University of California, Berkeley
R. W. Baker	MTR, Inc.
W. J. Koros	University of Texas, Austin
R. L. Riley	Separation Systems Technology, Inc.
D. Elyanow	Ionics, Inc.
L. Costa	Ionics, Inc.
K. Sims	Ionics, Inc.
T. Davis	Graver Water, Inc.
P. M. Gallagher	Alcan International, U.K.
W. Gudernatsch	Fraunhofer Institute, West Germany
A. L. Athayde	MTR, Inc.

GENERAL WORKSHOP HELD AT THE GORDON RESEARCH CONFERENCE

Attendee	Affiliation
W. Eykamp	University of California, Berkeley
R. W. Baker	MTR, Inc.
W. J. Koros	University of Texas, Austin
R. L. Riley	Separation Systems Technology, Inc.
H. Strathmann	Fraunhofer Institute, West Germany
E. L. Cussler	University of Minnesota
J. Beasley	Consultant
C. H. Lee	AMT
T. Lawford	EG&G Idaho
A. Allegreza	Millipore
L. Zeman	Millipore
G. Blytas	Shell Chemical Co.
D. Fain	Martin Marietta Energy Systems
J. D. Way	Oregon State University
K. Murphy	Permea - Monsanto
I. Roman	E. I. Du Pont de Nemours, Inc.
E. Sanders	Dow Chemical Corp.
G. Tkacik	Millipore
W. Robertson	PPG
R. L. Hapke	SRI International
J. Pellegrino	NIST
L. Costa	Ionics, Inc.
A. L. Athayde	MTR, Inc.

2.5 SECOND GROUP MEETING

The second group meeting was held during the Gordon Research Conference and was attended by all of the authors. The final format of each chapter was discussed and format revisions, based on comments from the expert workshops, were adopted.

2.6 JAPAN/REST OF THE WORLD SURVEY

This study contains a review of the state of the art of membrane science and technology in Japan, Europe and the rest of the world. Particular emphasis is placed on support of membrane research by foreign governments and sources of innovation in other countries. Two of the authors (Eykamp and Riley) visited Japan to collect information on membrane research in that country. Information on Europe was provided by Dr. Strathmann.

2.7 PRIORITIZATION OF RESEARCH NEEDS

The expert workshops identified over 100 research needs in membrane separations. Although these items had been rated in terms of importance and prospect of realization, they had been ranked within the individual sections of membrane technology. To facilitate the prioritization process, the research needs were condensed into a short list of 38 items, with the 5-7 highest ranked items selected from each of the individual sections.

The short list of research needs was submitted to the group of authors, who were asked to rank each of the items on the basis of energy-saving potential and other objectives related to DOE's mission.

2.8 PEER REVIEW

The report was submitted to a group of 10 reviewers selected by the DOE. Table 2-3 is a list of the reviewers. The reviewers comments, along with rebuttals or responses as appropriate, are presented in Appendix A.

Table 2-3. List of Peer Reviewers

<u>Name</u>	<u>Affiliation</u>
Dr. J. L. Anderson	Carnegie Mellon University
Dr. J. Henis	Monsanto
Dr. J. L. Humphrey	J.L. Humphrey and Associates
Dr. S.-T. Hwang	University of Cincinnati
Dr. N. N. Li	Allied Signal Corp.
Dr. S. L. Matson	Sepracor, Inc.
Dr. R. D. Noble	University of Colorado
Dr. M. C. Porter	M. C. Porter and Associates
Dr. D. L. Roberts	SRI International
Dr. S. A. Stern	Syracuse University

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CHAPTER THREE
INTRODUCTION
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CHAPTER THREE

INTRODUCTION

3.1 MEMBRANE PROCESSES

Seven major membrane processes are discussed in this report. They are listed in Table 3-1. There are four developed processes, microfiltration (MF), ultrafiltration (UF), reverse osmosis (RO), and electrodialysis (ED). These are all well established and the market is served by a number of experienced companies. The first three processes are related filtration techniques, in which a solution containing dissolved or suspended solutes is forced through a membrane filter. The solvent passes through the membrane; the solutes are retained.

Table 3-1. Membrane Technologies Addressed in This Report

	Process	Status
Developed technologies	Microfiltration Ultrafiltration Reverse Osmosis Electrodialysis	Well established unit processes. No major breakthroughs seem imminent
Developing technologies	Gas separation Pervaporation	A number of plants have been installed. Market size and number of applications served is expanding rapidly.
To-be-developed technologies	Facilitated transport	Major problems remain to be solved before industrial systems will be installed

Microfiltration, ultrafiltration, and reverse osmosis differ principally in the size of the particles separated by the membrane. Microfiltration is considered to refer to membranes that have pore diameters from 0.1 μm (1,000 \AA) to 10 μm . Microfiltration membranes are used to filter suspended particulates, bacteria or large colloids from solutions. Ultrafiltration refers to membranes having pore

diameters in the range 20-1,000 Å. Ultrafiltration membranes can be used to filter dissolved macromolecules, such as proteins, from solution. Typical applications of ultrafiltration membranes are concentrating proteins from milk whey, or recovery of colloidal paint particles from electrocoat paint rinse waters.

In the case of reverse osmosis, the membrane pores are so small, in the range of 5-20 Å in diameter, that they are within the range of the thermal motion of the polymer chains. The most widely accepted theory of reverse osmosis transport considers the membrane to have no permeant pores at all.¹ Reverse osmosis membranes are used to separate dissolved microsolute, such as salt, from water. The principal application of reverse osmosis is the production of drinking water from brackish groundwater, or the sea. Figure 3-1 shows the range of applicability of reverse osmosis, ultrafiltration, microfiltration and conventional filtration.

The fourth fully developed membrane process is electrodialysis, in which charged membranes are used to separate ions from aqueous solutions under the driving force of an electrical potential difference. The process utilizes an electrodialysis stack, built on the filter-press principle, and containing several hundred individual cells formed by a pair of anion and cation exchange membranes. The principal application of electrodialysis is the desalting of brackish groundwater. However, industrial use of the process in the food industry, for example to deionize cheese whey, is growing, as is its use in pollution-control applications. A schematic of the process is shown in Figure 3-2.

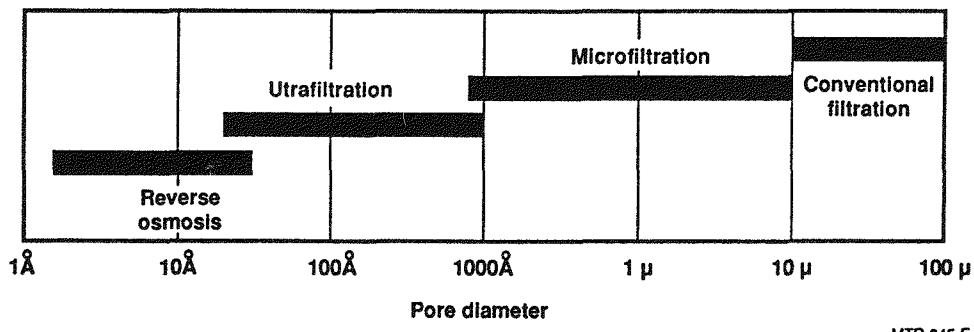
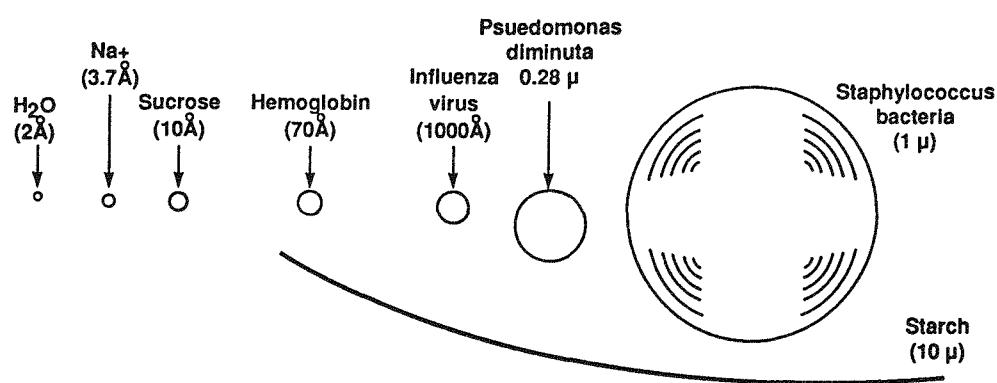


Figure 3-1. Reverse osmosis, ultrafiltration, microfiltration and conventional filtration are all related processes differing principally in the average pore diameter of the membrane filter. Reverse osmosis membranes are so dense that discrete pores do not exist. Transport in this case occurs via statistically distributed free volume areas. The relative size of different solutes removed by each class of membrane is illustrated in this schematic.

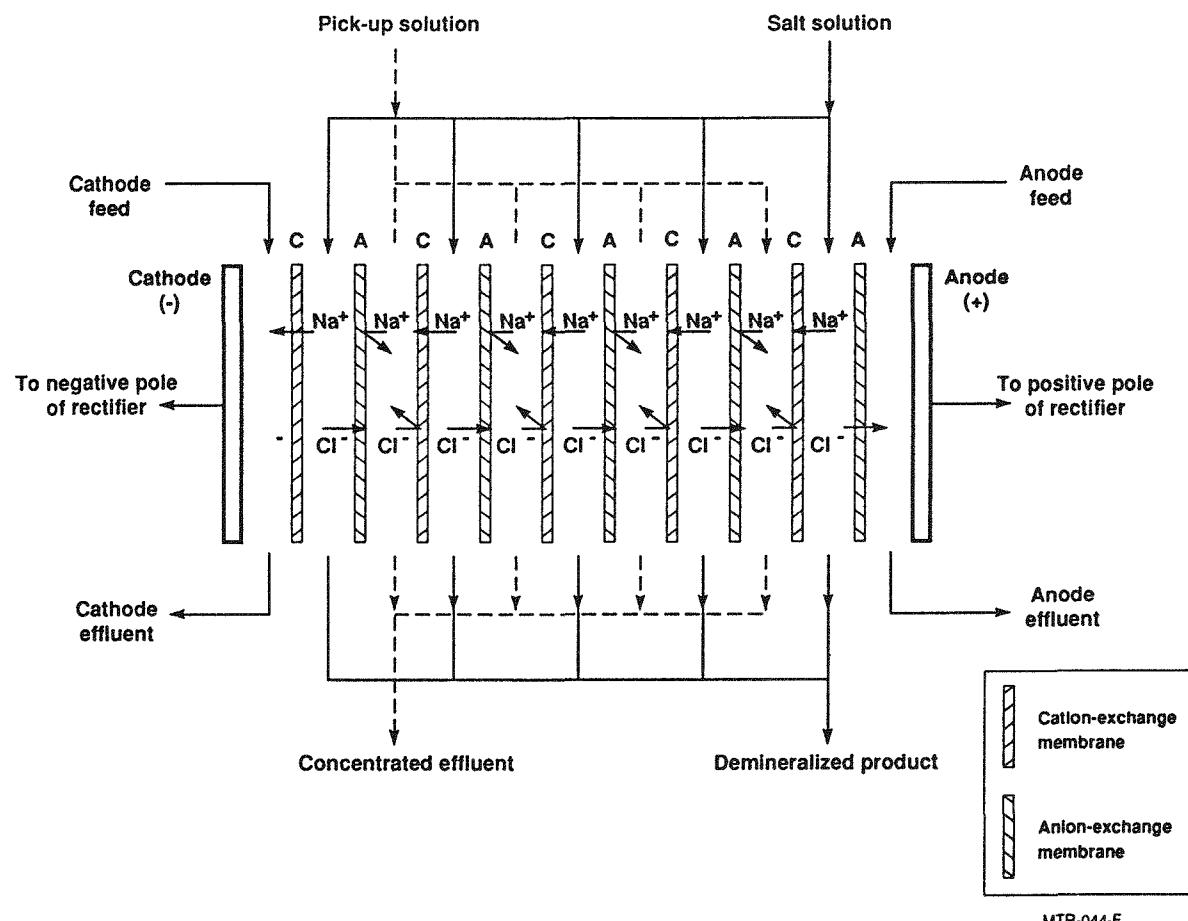
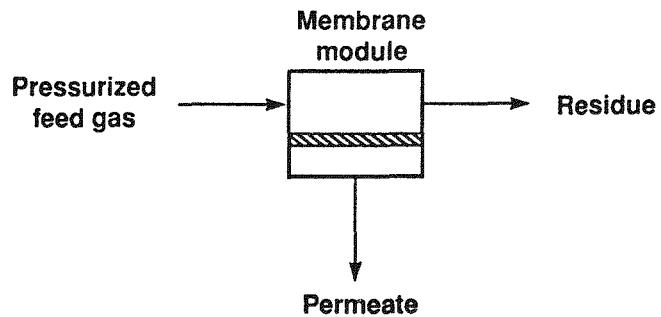


Figure 3-2. A schematic diagram of an electrodialysis process.

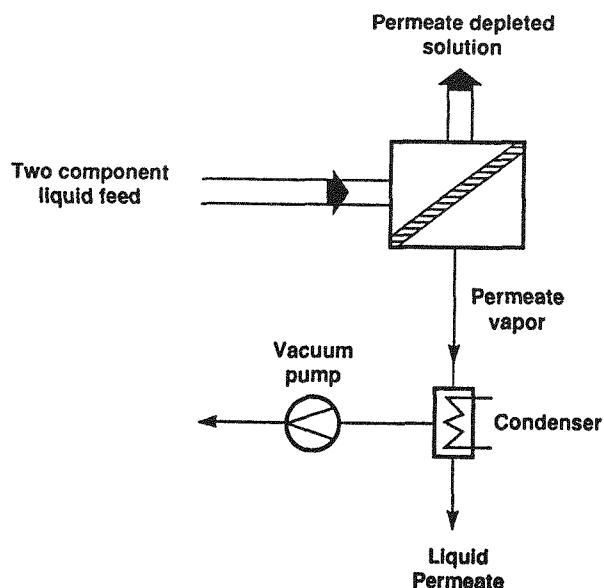
There are two developing processes: gas separation with polymer membranes and pervaporation. Gas separation with membranes is the more developed of the two techniques. At least 20 companies worldwide offer industrial, membrane-based gas separation systems for a variety of applications. Two companies currently offer industrial pervaporation systems. The potential for each process to capture a significant slice of the separations market is large. In gas separation, a mixed gas feed at an elevated pressure is passed across the surface of a membrane that is selectively permeable to one component of the feed. The membrane separation process produces a permeate enriched in the more permeable species and a residue enriched in the less permeable species. The process is illustrated in Figure 3-3. Major current applications are the separation of hydrogen from nitrogen, argon and methane in ammonia plants, the production of nitrogen from air and the separation of carbon dioxide from methane in natural gas operations. Gas separation is an area of considerable current research interest and it is expected that the number of applications will expand rapidly over the next few years.

Pervaporation is a relatively new process that has elements in common with reverse osmosis and gas separation. In pervaporation, a liquid mixture is placed in contact with one side of a membrane and the permeate is removed as a vapor from the other. The mass flux is brought about by maintaining the vapor pressure on the permeate side of the membrane lower than the partial pressure of the feed liquid. This partial pressure difference can be maintained in several ways. In the laboratory, a vacuum pump is used. Industrially, the low pressure is generated by cooling and condensing the permeate vapor. A schematic of a simple pervaporation process using a condenser to generate the permeate vacuum is shown in Figure 3-4. Currently, the only industrial application of pervaporation is the dehydration of organic solvents, in particular, the dehydration of 90-95% ethanol solutions, a difficult separation problem because of the ethanol-water azeotrope at 95% ethanol. However, pervaporation processes are being developed for the removal of dissolved organics from water and the separation of organic solvent mixtures. If the pervaporation of organic mixtures becomes commercial, it will replace distillation in a number of very large commercial applications.



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Figure 3-3. Schematic of a membrane gas separation process.



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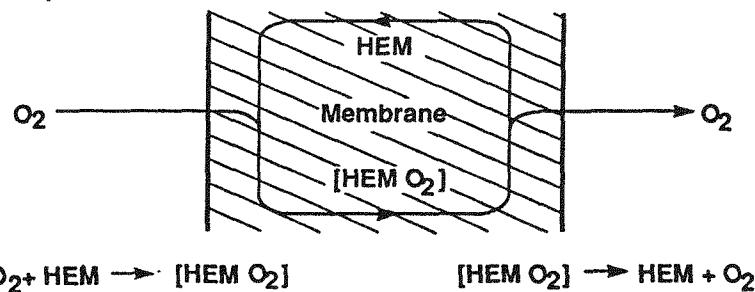
Figure 3-4. Schematic of a pervaporation process.

The final membrane process studied in the report is facilitated transport. This process falls under the heading of "to be developed" technology. Facilitated transport usually employs liquid membranes containing a complexing or carrier agent. The carrier agent reacts with one permeating component on the feed side of the membrane and then diffuses across the membrane to release the permeant on the product side of the membrane. The carrier agent is then reformed and diffuses back to the feed side of the membrane. The carrier agent thus acts as a shuttle to selectively transport one component from the feed to the product side of the membrane.

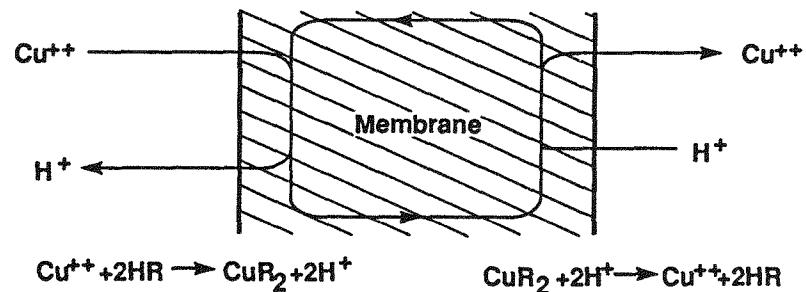
Facilitated transport membranes can be used to separate gases; membrane transport is then driven by a difference in the gas partial pressure across the membrane. Metal ions can also be selectively transported across a membrane, driven by a flow of hydrogen or hydroxyl ions in the other direction. This process is sometimes called coupled transport. Examples of facilitated transport processes for ion and gas transport are shown in Figure 3-5.

Because the facilitated transport process employs a reactive carrier species, very high membrane selectivities can be achieved. These selectivities are often far larger than the selectivities achieved by other membrane processes. This one fact has maintained interest in facilitated transport for the past 20 years. Yet no significant commercial applications exist or are likely to exist in the next decade. The principal problem is the physical instability of the liquid membrane and the chemical instability of the carrier agent.

Facilitated transport



Coupled transport



MTR-046-F

Figure 3-5. Schematic examples of facilitated transport of ions and gas. The gas-transport example shows the transport of O₂ across a membrane using hemoglobin as the carrier agent. The ion-transport example shows the transport of copper ions across the membrane using a liquid ion-exchange reagent as the carrier agent.

3.2 HISTORICAL DEVELOPMENT

Systematic studies of membrane phenomena can be traced to the eighteenth century philosopher scientists. The Abbé Nolet, for example, coined the word osmosis to describe permeation of water through a diaphragm in 1748. Through the nineteenth and early twentieth centuries, membranes had no industrial or commercial uses. However, membranes were used as laboratory tools to develop physical/chemical theories. For example, the measurements of solution osmotic pressure with membranes by Traube² and Pfeffer³ were used by van't Hoff in 1887⁴ to develop his limit law, explaining the behavior of ideal dilute solutions. This work led directly to the van't Hoff equation and the ideal equation of state of a perfect gas. The concept of a perfectly selective semipermeable membrane was also used by Maxwell and others at about the same time when developing the kinetic theory of gases.

Early investigators experimented with any type of diaphragm available to them, such as bladders of pigs, cattle or fish and sausage casings made of animal gut. In later work collodion (nitrocellulose) membranes were preferred, because they could be produced accurately by recipe methods. In 1906 Bechhold devised a technique to prepare nitrocellulose membranes of graded pore size, which he determined by a bubble-test method.⁵ Later workers, particularly Elford⁶, Zsigmondy and Bachman⁷, and Ferry⁸, improved on Bechhold's technique. By the early 1930s microporous collodion membranes were commercially available. During the next 20 years this early microfiltration membrane technology was expanded to other polymers, particularly cellulose acetate, and membranes found their first significant applications in the filtration of drinking water samples at the end of World War II. Drinking water supplies serving large communities in Germany and elsewhere in Europe had broken down and there was an urgent need for filters to test the water for safety. The research effort to develop these filters, sponsored by the U.S. Army, was later exploited by the Millipore Corporation, the first and largest microfiltration membrane producer.

By 1960, therefore, the elements of modern membrane science had been developed. But membranes were used in only a few laboratories and small, specialized industrial applications. There was no significant membrane industry and total sales of membranes for all applications probably did not exceed \$20 million per year. Membranes suffered from four problems that prohibited their widespread use: they were too unreliable, too slow, too unselective, and too expensive. Partial solutions to each of these problems have been developed during the last 30 years, and as a result there is a surge of interest in membrane-based separation techniques.

The seminal discovery that transformed membrane separation from a laboratory to an industrial process was the development, in the early 1960s, of the Loeb-Sourirajan process for making defect-free, high-flux, ultrathin reverse osmosis membranes.⁹ These membranes consist of an ultrathin, selective surface film supported on a microporous support that provides the mechanical strength. The first Loeb-Sourirajan membranes had fluxes 10 times higher than any membrane then available and made reverse osmosis a practical technology. The work of Loeb and Sourirajan, and the timely infusion of large sums of research dollars from the U.S. Department of Interior, Office of Saline Water (OSW), resulted in the commercialization of reverse osmosis and was a major factor in the development of ultrafiltration and microfiltration. The development of electrodialysis was also aided by OSW funding.

The 20-year period from 1960 to 1980 produced a tremendous change in the status of membrane technology. Building on the original Loeb-Sourirajan membrane technology, other processes were developed for making ultrathin, high-performance membranes. Using such processes, including interfacial polymerization or multilayer composite casting and coating, it is now possible to make membranes as thin as 0.1 μm or less. Methods of packaging membranes into spiral-wound, hollow-fine-fiber, capillary and plate-and-frame modules were also developed, and advances were made in improving membrane stability. As a result, by 1980 microfiltration, ultrafiltration, reverse osmosis and electrodialysis were all established processes with large plants installed around the world.

The principal development of the last 10 years has been the emergence of industrial membrane gas-separation processes. The first major development was the Monsanto Prism® membrane for hydrogen separation, in 1980.¹⁰ Within a few years, Dow was producing systems to separate nitrogen from air and Cynara and Separax were producing systems for the separation of carbon dioxide from methane. Gas-separation technology is evolving and expanding rapidly and further substantial growth will be seen in the 1990s. The final development of the 1980s was the introduction by GFT, a small German engineering company, of the first commercial pervaporation systems for dehydration of alcohol. By 1988, GFT had sold more than 100 plants. Many of these plants are small, but the technology has been demonstrated and a number of other major applications are at the pilot-plant scale.

3.3 THE FUTURE

In 1960, the dawn of modern membrane technology, the problems of membranes were selectivity, productivity/cost, and operational reliability. These problems remain the focus of membrane research today.

3.3.1 Selectivity

The problem of selectivity i.e., the ability of the membrane to make the required separation, has been essentially solved in some processes, but remains the key problem in others. For example, in 1960, no membranes were known with a high enough flux to make reverse osmosis an economically viable technology. The first Loeb-Sourirajan membranes, produced in 1960-63, had high fluxes and were able to remove 97-98% of the dissolved salt. This development made the process commercial. By the early 1970s, Riley, at Gulf General Atomic, had improved the salt-removal capability to 99.5%.¹¹ By the 1980s, Cadotte had produced interfacial composite membranes able to remove 99.8-99.9% of the dissolved salt.¹² Further improvements in the selectivity of reverse osmosis membranes are not required. Similarly, current microfiltration, ultrafiltration and electrodialysis membranes are generally able to perform the selective separation required of them. On the other hand, good membrane selectivity remains a generally unsolved problem in the

cases of gas separation and pervaporation. But here too, dramatic strides are being made. For example, the first commercial air-separation membranes used conventional polymers such as silicone rubber, ethylcellulose or poly-trimethylpentene, with oxygen/nitrogen selectivities in the range 2-4. The next generation of air-separation membranes now entering the marketplace uses polymers specifically designed for oxygen/nitrogen separation application. These membranes have selectivities of 6-8.¹³ More advanced materials, with even higher selectivities, have already been reported in the literature.

3.3.2 Productivity

It is usually possible to design a membrane system to perform a given separation. The problem is that a large, complex system, performing under energy-expensive operating conditions may be required. Thus, productivity, or separation performance per unit cost, is an issue in all membrane-separation processes.

There are a number of components to the problem of productivity and cost of membrane systems, including membrane materials, membrane configuration and membrane packaging efficiency. Membrane materials with higher intrinsic permeabilities clearly improve productivity. Similarly thinner, and thus higher-flux membranes, will reduce overall process costs, as will more economical ways of packaging these membranes into efficient modules. Having said this, there is a limit to the reduction in costs that can be achieved. For example, in a modern reverse-osmosis plant, membrane module costs generally represent only 25-35% of the total capital cost of the plant, and module replacement costs are not more than about 10% of the total operating cost. Even major reductions in membrane/module costs will, therefore, not change the economics of the reverse osmosis process dramatically. In the case of reverse osmosis, cost reductions may be more easily achieved by improving nonmembrane parts of the process, for example, the water pretreatment system. However, in some processes such as microfiltration, membrane and module costs are more than 50% of the operating cost. Cost reductions in the membrane/module area would, therefore, be useful in these processes.

3.3.3 Operational Reliability

Operational reliability is the third and the most generally significant problem in membrane processes. The causes of reliability problems vary from process to process. Fouling is a critical factor in ultrafiltration and microfiltration and therefore dominates the entire membrane operation. Fouling is also a major factor in reverse osmosis. In gas separation, fouling is usually not a problem and only minimal pretreatment of the feed stream is required. On the other hand, in a typical membrane gas-separation process, it is only necessary to develop one defect per square meter of membrane to essentially destroy the efficiency of the process. The ability to make, and maintain, defect-free membranes is, therefore, a key issue in gas separation.

Another factor that leads to operational unreliability is poor membrane stability. In facilitated-transport membranes, instability is such a problem that the process has never become commercial. Membrane instability has also proved to be a major problem area in reverse osmosis, gas separation and pervaporation.

There is no panacea for system reliability. The solution usually appears to be a combination of a number of factors, such as better membrane materials, better module designs, improved cleaning and antifouling procedures, and better process designs. A summary table outlining the relative magnitude of these problem areas for the seven membrane technologies discussed in this report is shown in Table 3-2 below.

Table 3-2. Development Status of Current Membrane Technologies

Process	Problems			Comments
	Major	Minor	Mostly solved	
Micro-filtration	Reliability (fouling)	Cost	Selectivity	Better fouling control could improve membrane lifetime significantly.
Ultra-filtration	Reliability (fouling)	Cost	Selectivity	Fouling remains the principal operational problem of ultrafiltration. Current fouling control techniques are a substantial portion of process costs.
Reverse osmosis	Reliability	Selectivity	Cost	Incremental improvements in membrane and process design will gradually reduce costs.
Electro-dialysis	Fouling Temperature stability	Cost	Selectivity Reliability	Process reliability and selectivity are adequate for current uses. Improvements could lead to cost reduction, especially in newer applications.
Gas separation	Selectivity Flux	Cost	Reliability	Membrane selectivity is the principal problem in many gas separation systems. Higher permeation rates would help to reduce costs.
Pervaporation	Selectivity Reliability	Cost	-	Membrane selectivities must be improved and systems developed that can reliably operate with organic solvent feeds before major new applications are commercialized.
Coupled and Facilitated Transport	Reliability (membrane stability)		-	Membrane stability is an unsolved problem. It must be solved before this process can be considered for commercial applications.

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CHAPTER FOUR
GOVERNMENT SUPPORT OF MEMBRANE RESEARCH

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CHAPTER FOUR

GOVERNMENT SUPPORT OF MEMBRANE RESEARCH

4.1 OVERVIEW

Membrane science originated in Europe and many of the fundamental laws and equations of membrane science bear the names of European scientists, Graham's Law, Fick's Law, the van't Hoff equation, the Donnan effect and so on. European dominance of the field lasted until the early 1950s, when a new membrane industry, centered in the United States, began. Federal research support played a critical role in the early growth of this industry. Millipore, now the world's largest microfiltration company, got its start out of a U.S. Army contract to develop membrane filters. The reverse-osmosis and electrodialysis industries received even more significant levels of support from the Office of Saline Water from 1960 to 1975. Poor drinking-water quality in the southern and southwestern states, plus the possibility of increasing water supplies to arid regions, were seen as problems that could be addressed by the newly emerging membrane technology. Despite the fact that no membrane industry as such existed, the U.S. Government made a far-sighted commitment to the new technology. As a result, the industry received an average of between \$20-40 million per year (in 1990 dollars) for membrane research over a period of 15 years.

During this "Golden Age", hollow fibers, spiral-wound modules, asymmetric membranes, thin-film composites and all the other basic components of current membrane technology were developed. Not only did reverse osmosis and electrodialysis research rely almost completely on the flow of Federal research monies, but the ultrafiltration industry, and to a lesser extent the microfiltration industry, also received considerable assistance from the fallout of this support. Finally a significant invention, the spiral-wound module, tightly patented and licensed gratis by the Government to U.S. companies, was decisive in maintaining U.S. dominance over reverse-osmosis markets through the 1970s. Few outside the industry appreciate the importance of these patents in blocking non-U.S. firms.

In 1975 the Office of Saline Water closed and there was a substantial reduction in the level of Federal membrane research support, from \$40 million per year (1990 dollars) to the present level of \$10-11 million. The demise of the Office of Saline Water coincided with a surge of interest in the membrane industry in Japan and Europe.

In Europe and Japan there is a significant amount of government research support to academic institutions and to private industry. Furthermore, the level of support appears to be growing. The approximate levels of support in the United States, Japan and Europe are summarized in Table 4-1.

Table 4-1. Government Membrane Support

		<u>Level of Support (\$ millions/year)</u>
<u>United States</u>	DOE: Office of Industrial Programs	1.5
	Office of Basic Energy Research	1.0
	Office of Fossil Energy	1.0
	SBIR Programs	1.0
	NSF	4.0
	EPA	1.5
	NASA	0.5
	DOD	<u>0.5</u>
	Total	11.0
<u>Japan</u>	Ministry of Education: Membrane Support to Universities	2.0 (est.)
	MITI: Basic Industries Bureau	2.0 (est.)
	AIST - Jisedai Project	2.0 (est.)
	Aqua Renaissance '90	5.0
	WRPC	6.0
	NEDO	<u>2.0</u>
	Total	19.0
<u>Europe</u>	National Programs for University Support	10.0 (est.)
	National Membrane Programs: Holland	2.0
	U.K.	1.5
	Italy	2.5
	EEC (BRITE) Program	<u>4.0</u> (est.)
	Total	20.0

The numbers in this table should be treated with caution. The U.S. numbers are fairly reliable, as are the numbers for the foreign, individually designated programs. Numbers labeled "estimated" are however, just that and are not reliable to better than 30%. Currently it appears that total U.S. Government funding for membrane research is approximately \$10-11 million per year, compared to approximately \$19 million per year in Japan and \$20 million per year in Europe.

In part because of the significant amount of research support that Japanese and European companies have received, the dominant position that the U.S. membrane industry enjoyed in world markets until 1980 has been eroded. Japanese companies have largely recaptured their domestic markets in reverse-osmosis, ultrafiltration and electrodialysis. Japanese companies now compete strongly with U.S. suppliers in the areas of reverse osmosis and electrodialysis in the Middle East. In the U.S. and Europe, Japanese companies have been less successful. After failing to establish their own subsidiaries in the U.S., they are beginning to enter the market by acquiring U.S. companies. For example, Nitto Denko, a major Japanese reverse-osmosis and ultrafiltration company, recently acquired Hydranautics, the third or fourth biggest U.S. reverse-osmosis company. Toray Industries, another large Japanese firm, has also tried to acquire a U.S. reverse osmosis company.

European companies have been less successful than the Japanese in capturing their home markets and in competing overseas. There are a number of significant European membrane companies, but they have not succeeded in displacing American companies from their dominant position in ultrafiltration, reverse osmosis and electrodialysis.

In gas separation and pervaporation, which represent the emerging membrane industry, the commercial markets are still fluid. In gas separation, U.S. companies are ahead. In pervaporation, European and Japanese companies lead, with the United States trailing significantly behind. The extent of future government support to the universities and to industry will have a significant effect on the final U.S. position in these technologies.

4.2 U.S. GOVERNMENT SUPPORTED MEMBRANE RESEARCH

The current level of support of membrane-related research by the U.S. Government is of the order of \$11 million annually. The Department of Energy, which funds energy-related membrane research and development, is one of the significant sources of U.S. Government support. The National Science Foundation is the other major source of support, particularly for academic institutions and others carrying out fundamental research in membrane science. Other sources of funding include the Environmental Protection Agency, the Department of Defense, the National Aeronautics and Space Administration and the Department of Agriculture, which support research and development of membrane separation systems that are relevant to the specific mission of the department or agency.

4.2.1 Department of Energy

The U.S. Department of Energy supports membrane separations research and development via several programs. The emphasis in all of these programs is on devices and processes that have the potential for high energy savings. The current level of funding of the DOE's membrane research and development programs is between \$4.3-4.5 million annually. The most significant of these programs is the Industrial Energy Conservation Program. This program is sponsored by the Division of Improved Energy Productivity of the Office of Industrial Programs in the Office of Conservation and Renewable Energy.

4.2.1.1 Office of Industrial Programs/Industrial Energy Conservation Program

The mission of the Office of Industrial Programs is to increase the end-use energy efficiency of industrial operations. The Industrial Energy Conservation Program, administered by this office, is designed to fund research and development of high-risk, innovative technologies to increase the energy efficiency of industrial operations. Federal funding can accelerate industry's acceptance of a new technology by alleviating some of the risk associated with commercialization. Research and development of membrane separation processes for the paper, textile, chemical and food-processing industries have been funded by this program since 1983. The current level of support is of the order \$1.5 million per year. Table 4-2 contains a list of the specific projects and the contractors.

Table 4-2. Membrane R&D Funded through the Office of Industrial Programs since 1983

Contractor	Topic
Air Products & Chemicals, Inc.	Active transport membranes
Alcoa Separations	Catalytic membrane reactor
Allied-Signal Corp.	Fluorinated membranes
Allied-Signal Corp.	Membranes for petrochemical applications with large energy savings
American Crystal Sugar Co. and the Beet Sugar Develop. Found.	Concentrating hot, weak sugar-beet juice
Bend Research, Inc.	Membrane-based industrial air dryer
Bend Research, Inc.	Membrane separation system for the corn sweetener industry
Carre, Inc.	Dynamic membranes to reclaim hot dye rinse water
EG&G, Inc.	Polyphosphazene membranes
EG&G, Inc.	Assessment of membrane separations in the food industry
Filmtec Corp.	Temperature-resistant, spiral-wound elements
HPD, Inc.	Electrolysis of Kraft Black Liquor
Ionics, Inc.	An electro-osmotic membrane process
Mavdil Corp.	Membrane for concentrating high solubles in water from corn wet milling
Membrane Technology & Research, Inc.	Removal of heat and solvents from industrial drying processes
National Food Processors Assn.	Develop energy-efficient separation, concentration and drying processes for food products

Table 4-2 continued

Contractor	Topic
National Food Processors Assn.	Hyperfiltration as an energy conservation technique for the renovation and recycle of hot, empty container wash water
Physical Sciences, Inc.	Reduced energy consumption for the production of chlorine and caustic soda
SRI International, Inc.	Piezoelectric membranes
SRI International, Inc.	Hybrid membrane systems
State University of New York	Energy-efficient, high-crystalline, ion-exchange membranes for the separation of organic liquids
State University of New York	Membrane dehydration process for producing high grade alcohols
University of Maine	Ultrafiltration of Kraft Black Liquor
University of Wisconsin	Colloid-chemical approach to the design of phosphate-ordered ceramic membranes
UOP, Inc.	A membrane oxygen-enrichment system

4.2.1.2 Office of Energy Research/Division of Chemical Sciences

The Division of Chemical Sciences of the Office of Basic Energy Sciences in the Office of Energy Research funds fundamental research into membrane materials and membrane transport phenomena. The objective of this support is to add to the available knowledge regarding membrane separations. The funds are primarily directed towards research at universities and the National Laboratories. The Division of Chemical Sciences spends \$500,000 per year on membrane-specific research and another \$500,000 per year on peripheral research fundamental to the understanding of membrane transport. Some industrial

research is also supported, but is administered through the Small Business Innovative Research Program (SBIR). Table 4-3 is a list of typical projects supported by the Division of Chemical Sciences.

Table 4-3. Membrane R&D Funded through the Division of Chemical Sciences

Contractor	Topic
Brigham Young University	Novel macrocyclic carriers for proton-coupled liquid-membrane transport
Lehigh University	Perforated monolayers
University of Oklahoma	A study of micellar-enhanced ultrafiltration
Syracuse University	Mechanisms of gas permeation through polymer membranes
University of Texas	Synthesis and analysis of novel polymers with potential for providing both high permselectivity and permeability in gas separation applications
Texas Tech University	Metal ion complexation by ionizable crown ethers

4.2.1.3 Office of Energy Research/Division of Advanced Energy Projects

The Division of Advanced Energy Projects within the Office of Energy Research complements the role of the Division of Chemical Sciences. Most of the projects supported involve exploratory research on novel concepts related to energy. The typical project has both very high risk and high payoff potential, and consists of concepts that are too early to qualify for funding by other Department of Energy programs. The support is sufficient to establish the scientific feasibility and economic viability of the project. The developers are then encouraged to pursue alternative sources of funding to complete the commercialization of the technology. The Division does not support ongoing, evolutionary research. Table 4-4 is a list of projects supported by the Division of Advanced Energy Projects during the past five years. At present, the Division is funding one membrane project on the separation of azeotropes by pervaporation at a level of \$150,000 per year.

**Table 4-4. Membrane R&D Funded through the
Division of Advanced Energy Projects since 1983**

Contractor	Topic
Bend Research, Inc.	The continuous membrane column; a low energy alternative to distillation
Bend Research, Inc.	Liquid membranes for the production of oxygen-enriched air
Membrane Technology & Research, Inc.	Pervaporation: A low-energy alternative to distillation
Membrane Technology & Research, Inc.	Separation of organic azeotropic mixtures by pervaporation
Portland State University	Thin-film composite membranes for artificial photosynthesis

4.2.1.4 Office of Fossil Energy

The Office of Fossil Energy supports research and development related to improving the energy efficiency of fossil-fuel production and use. The projects are typically administered through the Morgantown and Pittsburgh Energy and Technology Centers (METC & PETC). Membrane projects related to improved combustion processes and fuel and flue-gas cleanup are supported by the Gas Stream Cleanup and Gasification programs at METC and by the Flue Gas Cleanup program at PETC. The support for these programs amounts to about \$1.0 million per year. Representative research projects are listed in Table 4-5.

Table 4-5. Membrane R&D Funded through the Office of Fossil Energy since 1983

Contractor	Topic
Air Products & Chemicals, Inc.	High-temperature, facilitated-transport membranes
Alcoa Separations	Alumina membrane for high temperature separations
California Institute of Technology	Silica membranes for hydrogen separation
Jet Propulsion Laboratory	Zirconia cell oxygen source
Membrane Technology & Research, Inc.	Low-cost hydrogen/Novel membrane technology for hydrogen separation from synthesis gas
Membrane Technology & Research, Inc.	Development of a membrane SO _X /NO _X treatment system
METC (in-house)	Ceramic membrane development
National Institute for Standards & Technology	Gas separation using ion-exchange membranes
Oak Ridge National Laboratory	Gas separation using inorganic membranes
SRI International	Catalytic membrane development
SRI/PPG Industries	Development of a hollow fiber silica membrane
Worcester Polytech. Institute	Catalytic membrane development

4.2.1.5 Small Business Innovative Research Program

The Small Business Innovative Research Program (SBIR) was initiated by Congress in 1982 to stimulate technological innovation in the private sector and strengthen the role of small business in meeting Federal research and development needs. A greater return on investment from Federally funded research as well as increased commercial application are the other expected benefits from this program. The program consists of three phases and is open

only to small businesses. Phase I is typically a six-month feasibility study with funding up to \$50,000. If approved for follow-on funding, the project enters a two-year Phase II stage, of further development and scale-up, with support of up to \$500,000. A final non-funded stage, Phase III, consists of commercial or third-party sponsorship of the technology and represents the entry of the technology into the marketplace.

This program encompasses topics of interest to a number of subdivisions of the Department of Energy, including the Office of Fossil Energy (METC & PETC), the Office of Energy Research and the Office of Conservation and Renewable Energy. During 1989, the DOE-SBIR program supported two Phase II projects and five Phase I projects, totalling \$750,000 per year. Table 4-6 contains a list of the projects that have been supported under this program.

Table 4-6. Membrane-related SBIR Projects since 1983

Year initiated	Phase I II	Contractor	Topic
1983	X	Membrane Technology & Research, Inc.	Novel liquid ion-exchange extraction process
1983	X X	Bend Research, Inc.	Concentration of synfuel process condensates by reverse osmosis
1983	X X	Bend Research, Inc.	Solvent-swollen membranes for the removal of hydrogen sulfide and carbon monoxide from coal gases
1984	X X	Bend Research, Inc.	Thin-film composite gas separation membranes prepared by interfacial polymerization
1984	X	Membrane Technology & Research, Inc.	Improved coupled transport membranes
1985	X	Magna-Seal, Inc.	Perfluorinated crosslinked ion-exchange membranes
1985	X	Membrane Technology & Research, Inc.	Plasma-coated composite membranes
1985	X	Merix Corp.	Improved hydrogen separation membranes
1985	X	Process Research & Development, Inc.	Separation of oxygen from air using amine-manganese complexes in membranes
1985	X X	Bend Research, Inc.	A membrane-based process for flue gas desulfurization
1986	X	Foster-Miller, Inc.	A high-performance gas separation membrane
1987	X X	Bend Research, Inc.	Novel high-flux antifouling membrane coatings
1987	X X	Bend Research, Inc.	High-flux, high-selectivity cyclodextrin membranes
1988	X X	Spire Corp.	Novel electrically conductive membranes for enhanced chemical separation

Table 4-6. continued

Year initiated	Phase I II	Contractor	Topic
1988	X	Texas Research Institute	Synthesis of new polypyrrones and their evaluation as gas separation membranes
1988	X X	CeraMem Corp.	A ceramic membrane for gas separations
1989	X	Cape Cod Research, Inc.	A molecular recognition membrane
1989	X	CeraMem Corp.	Low-cost ceramic support for high-temperature gas separation membranes
1989	X	CeraMem Corp.	Low-cost ceramic ultrafiltration membrane module
1989	X	KSE, Inc.	Chlorine-resistant reverse osmosis membrane
1989	X	Coury & Associates	Novel surface modification approach to enhance the flux/selectivity of polymeric membranes
1989	X	Membrane Technology & Research, Inc.	Membranes for a flue gas treatment process
1989	X	Membrane Technology & Research, Inc.	Novel membranes for natural gas liquids recovery

4.2.2 National Science Foundation

The National Science Foundation (NSF) supports fundamental research in membrane separations both at universities and in industry through research grants and SBIR awards. The level of funding of the NSF membrane research program is comparable to that of the DOE (\$4 million dollars annually) although the mission of these two programs is quite different. Unlike the DOE, which funds energy-related research with an emphasis on the development of viable technology, the NSF funds exploratory research and fundamental studies that increase the understanding of the transport phenomena in membranes.

The Division of Chemical and Thermal Systems currently funds 50-60 projects per year in membrane-related research. The average project receives about \$60,000 per year and the total value of the program is \$3.5 million. The projects funded are fundamental studies of the basics of membrane science and membrane materials. Although this work is important to the understanding and use of membrane separation processes, not all of it is relevant to the energy conservation issues addressed in this report.

A new program jointly administered by the Divisions of Life Sciences and Chemical and Thermal Systems, has been set up to fund membrane-related research in biotechnology at a rate of \$500,000 per year. Most projects will receive \$60,000 per year, with one or two group awards of \$200,000 per year.

Research in polymer and inorganic materials funded by the Division of Materials Research also contributes to the body of knowledge on membranes.

4.2.3 Environmental Protection Agency

The Environmental Protection Agency (EPA) supports membrane separation system research and development primarily through the SBIR and the Superfund Innovative Technology Evaluation (SITE) programs. The research funded is related to EPA's mission of reduction, control and elimination of hazardous wastes discharged to the environment. The current level of funding for membrane-related research is of the order of \$1.3 million per year.

The SBIR program currently supports projects investigating the use of membranes for the removal of organic vapors from air and the removal of volatile organic contaminants from aqueous streams. The present level of funding in the SBIR program is on the order of \$750,000 per year.

The SITE program was set up as part of the Superfund Amendments and Reauthorization Act of 1986 (SARA). It is administered by the EPA's Office of Solid Waste and Emergency Response and the Office of Research and Development. The Emerging Technologies Program (ETP), a component program of SITE, is designed to assist private developers in commercializing alternative technologies

for site remediation. The research projects funded through the ETP are typically bench- and pilot-scale testing of new technologies and are funded at a level of \$150,000 per year. The three membrane-related projects being funded through the ETP are listed in Table 4-7.

Table 4-7. Membrane R&D Funded through the Emerging Technologies Program

Contractor	Topic
Atomic Energy of Canada Ltd.	Ultrafiltration of metal/chelate complexes from water
Membrane Technology & Research, Inc.	Removal of organic vapor from contaminated air streams using a membrane process
Wastewater Technology Center	Cross-flow pervaporation system for the removal of VOC's from aqueous wastes

4.2.4 Department of Defense

The Department of Defense (DOD) funds a small number of membrane separations research projects through its SBIR program. These projects address specific strategic and tactical needs of the DOD, but are also applicable to industrial separations. Examples of such research are:

- Chlorine-resistant hollow-fiber reverse osmosis elements for portable desalination units
- Membranes for on-board water generation from vehicular exhausts
- Membrane oxygen extraction units for providing breathable air in chemically contaminated environments
- Polymeric and liquid membranes for the extraction of oxygen from seawater

As the type of research and level of support is governed by the current needs of the DOD, there is no specific program for membrane research. Consequently, funding is small and intermittent.

4.2.5 National Aeronautics and Space Administration

The National Aeronautics and Space Administration (NASA) has funded a few membrane-related projects through the SBIR program. These projects are oriented toward NASA's mission in space and consist of new technology for life support systems in space. The areas of research supported are:

- Membrane systems for removal and concentration of carbon dioxide in the space vehicle cabin atmosphere
- Membrane systems for water recovery and purification

Since the type of research and level of support is governed by the current needs of NASA, there is no specific program for membrane research. Consequently, funding is small and intermittent.

4.3 JAPANESE GOVERNMENT SUPPORTED MEMBRANE RESEARCH

Although the dates of origin of the membrane industries in Japan and the U.S. differ by about 20 years, in many ways the experiences of the two countries are similar. The Japanese government continues to support a large research effort in membranes that began in the 1970s. A number of programs will begin to expire in the early 1990s, but will undoubtedly be replaced by others, although their size may decrease and their focus change. A reduction in government support would reflect the current size and status of the Japanese membrane industry. Some leading Japanese companies no longer participate directly in Government-sponsored programs. They prefer to support research efforts with their own funds, in this way maintaining an edge over their competition. Having said this, the total level of Government membrane research support is currently twice the U.S. Government level.

Japan sponsors a variety of programs that support membrane research and development. A few are direct; most are indirect. The Ministry of Education, for example, does not have a membrane program *per se*, but membrane programs are included in the support of educational research. Aqua Renaissance '90, an agency of the Ministry of International Trade and Industry (MITI), supports work

on membranes as a means to achieve its goals in the area of water re-use. Other agencies also support membrane research and development as an opportunity to develop domestic products that will displace foreign imports and will ultimately be exported to world markets.

4.3.1 Ministry of Education

Academic research is sponsored by general grants to faculty, and by specific research programs with relevance to the membrane field. Ministry of Education programs are said to be primarily for the training of students, with little regard for the utility of the research in the near term. Pervaporation membrane research has been a particularly active area recently. The general level of this support is estimated at \$2 million annually.

4.3.2 Ministry of International Trade and Industry (MITI)

MITI sponsors research and development projects thought to have medium-term practical significance. Several membrane-related projects are included in the program. Some of the agencies and departments of MITI known to be sponsoring membrane work are listed below.

4.3.2.1 Basic Industries Bureau

This agency sponsors a project on membrane dehydration of alcohol (dehydration of azeotropes). The program began when GFT started selling pervaporation plants in Japan. Many separations are potential candidates for pervaporation technology. The program's goal is to develop superior technology. Its main focus has been on membranes, particularly those derived from chitosan, to make water-permeable dehydration membranes. Recently, three companies, Sasakura Engineering, Tokuyama Soda and Kuraray, announced that they had independently developed chitosan-based pervaporation membranes, whose properties are said to be competitive with GFT membranes. Details have not yet been revealed, although some of this work is now beginning to appear in the U.S. patent literature.

4.3.2.2 Agency of Industrial Science and Technology (AIST)

AIST is responsible for the National Laboratories, two of which have active membrane programs. The Government Industrial Research Institute (Osaka) is often mentioned in reports of membrane research. Programs are also under way at the National Chemical Laboratory for Industry (Tsukuba). AIST also conducts a project for revolutionary basic technologies, formally known as Research and Development Project of Basic Technologies for Future Industries, popularly known as the Jisedai Project. One of fourteen categories targeted for development is "Synthetic Membranes for New Separation Technology." Included are efforts to develop high-performance pervaporation and gas-separation membranes. This work is the responsibility of National Chemical Laboratory for Industry (Tsukuba), and has been performed at the Research Institute for Polymers and Textiles (AIST), the Industrial Products Research Institute (AIST), and at the Research Association for Basic Polymer Technology, an organization of 10 private companies and two universities.

Another AIST-sponsored project is the National Research and Development Program. Nine projects considered particularly important and urgent for the nation are under development. One of these is the New Water Treatment Program, known generally as Aqua Renaissance '90. The annual budget of the membrane program is in the region of \$4-5 million. This project is aimed at developing new ways to treat wastewater from a variety of sources (municipal, starch processing, etc.) in the Japanese context. One very important consideration in any Japanese waste-treatment facility is the plant footprint. Land in Japan is at a premium, so conventional secondary sewage treatment was eliminated at the outset of Aqua Renaissance '90 as requiring too much land. Membranes fit well into plans to build a new type of waste-treatment facility. Japan's lack of indigenous fuel also makes the production of methane from its wastes attractive. Thus the combination of anaerobic digestion and membrane concentration looked particularly attractive. The effort is funded at a level high enough to work out the problems and try the needed equipment. Whether this work will result in a new way of treating wastes remains to be seen. What is obvious is that the state of the membrane art generally has been advanced significantly as a result of the program.

The Aqua Renaissance '90 idea is not a novel concept. Dorr-Oliver worked on essentially the same approach for many years. They did not have the resources to solve all the problems and achieve commercial success. The problem proved too big and too complex for that one company to solve. If the project is a significant success, Japan stands to gain substantial external markets, because wastewater treatment is a ubiquitous problem. Many large cities throughout the world would be interested in replacing their existing sewage-treatment plants with high-efficiency, low-land-use alternatives.

4.3.2.3 Water Re-use Promotion Center (WRPC)

The WRPC is an incorporated foundation, chartered by, and partially funded by, MITI. It was set up in 1980 to promote water saving. Its activities involve desalination, water re-use, training and performance testing of membrane systems. It lists approximately 100 members, including local government and water authorities, engineering companies, manufacturing companies, banks and insurance companies. It has about 20 permanent employees and about 33 more on temporary assignment from their employers. These people are paid by WRPC and do training assignments as well as assessments sponsored by Japan International Cooperation Agency, usually as part of Japan's foreign aid program. The annual budget is approximately \$6 million. Major membrane-related projects conducted by WRPC in the year ending March, 1988, included:

- Experiments for establishing seawater desalination technology by reverse osmosis.
- Using solar cells to power reverse osmosis desalination systems.
- Electrodialysis for seawater desalting utilizing solar cells.
- Experiments for establishing a new technology for ultra-pure water production.
- Experiments on removal of malodor and color using activated carbon fiber.
- Studies on effective use of industrial water.

4.3.2.4 New Energy Development Organization (NEDO)

NEDO is an MITI-funded foundation established in 1980. Its charter is to consider alternatives to petroleum for energy supply. Recently, NEDO activities have been enlarged to involve all industrial technology. One of NEDO's programs, the Alcohol Biomass Energy Program, contains a project for development of membranes to maintain high densities of methanogenic bacteria, and development of modules for employing them. Their interest extends beyond this project to the broader area of water re-use.

4.3.3 Ministry of Agriculture, Forestry and Fisheries

This ministry is active in the membrane area through promotion of the use of membranes, particularly reverse osmosis and ultrafiltration membranes, in the food industry. There is a current program on chemical conversion of biomass involving membranes and a completed project on wastewater treatment for the food industry.

4.4 EUROPEAN GOVERNMENT SUPPORTED MEMBRANE RESEARCH

Europe is a significant importer of industrial membrane separation equipment and a major market for U.S. industrial membrane manufacturers, particularly microfiltration and ultrafiltration equipment suppliers. Pall, Millipore and Koch Membrane Systems all derive significant benefit from their activities in Europe. There are also strong European companies, however, in the areas in which the Americans have traditionally been most successful (DDS, Sartorius, PCI, S & S, Rhone Poulenc). The U.S. position could change.

In the emerging field of pervaporation, GFT, the German subsidiary of a French company, is the undisputed world leader at present.

4.4.1 European National Programs

European membrane research groups receive support from their own national governments and from the multinational groupings such as the European Economic Community (EEC). The amount of support given by national programs is difficult to track because it is hidden in the general funds given to the universities. A recent survey by the European Membrane Society identified a total of 79 universities and institutes where there were significant membrane science and technology research programs. Some of these groups are very large, for example, the groups at the University of Twente (Holland), GKSS Geesthacht (West Germany) and the Fraunhofer Institute of Stuttgart (West Germany). These groups each have more than 20 research students and staff and budgets of several million dollars. Other groups are undoubtedly smaller and may consist of a professor and one or two students, with a budget of \$100,000 or less. We believe that an estimate of \$10 million disbursed by various national Government Ministries of Education and Science to support membrane research in academia is conservative. This estimate is in accord with an intuitive sense of the relative size of the European and American academic interests in membranes. In addition to this general support, there are some specific national membrane programs aimed at industry and academic groups. The more important of these groups are discussed below.

- The Dutch Innovative Research Program on Membrane Technology. This project funded over seven years at \$2 million per year is aimed at producing new membranes for gas separation, pervaporation and ultrafiltration. Membrane fouling is another topic area.
- The United Kingdom Science and Engineering Council Program. This five-year program has an annual budget of \$1.5 million. Research is aimed at a wide range of basic and applied membrane topics.
- The Italian National Project in Fine Chemicals. This program, with a budget of \$2.5 million annually, supports 20 academic and industrial teams working in the membrane area.

4.4.2 EEC-Funded Membrane Research

In addition to the national membrane programs, membrane-research support is available through the EEC. The most important program to the membrane community is the Basic Research in Industrial Technologies for Europe (BRITE) program. The BRITE program is now in its second term. Within select research areas, projects within the Community may be subsidized up to 50%. All projects must have a sponsor in at least two member states, one of which must be industrial. Membranes were one of the areas selected for particular emphasis. The countries participating are France (F), the Netherlands (NL), the United Kingdom (UK), Italy (I), West Germany (D), Denmark (DK) and Spain (E). Amongst the topics funded were:

- Gas separation membranes for upgrading methane containing gases to pipeline quality. [Gerth (F) and Nederlandsse Gasunie (NL).]
- Gas separation membranes for separation of CO₂ and H₂S from natural gas. [Akzo (NL) and Elf Aquitaine (F) and University of Twente (NL).]
- Development of cross-flow microfiltration membranes for the biotechnology industry. [Tech Sep (F) and Advanced Protein Products (UK) and University of Loughborough (UK).]
- Development of inorganic and ceramic membranes for gas separations. [Eniricerche SPA (I) and Enichem (I), Harwell Laboratory (UK), Esmill Water Systems (NL), Hoogovens Groep (NL) and ECN (NL).]
- Application of membranes to the textile industry. [Separem (I), Peignage D'Auchel (F), Texilia (I), Fraunhofer Institute (D) and University of Calabria (I).]
- Integrated ultrafiltration and microfiltration membrane processes. [DDS (DK), Soc. Lyonnaise des Eaux (F), University College Wales (UK), Technical University of Denmark (DK) and Imperial College, London (UK).]
- The use of membranes to treat olive oil wastewater. [Inst. Ricerche Breda (I), Separem SPA (I), Labein (E), Pridesa (E) and Centro Richerche Bonomo (I).]
- Acid-stable pervaporation membranes. [BP Chemicals (UK), GFT (D), RWTH (D), University of Twente (NL) and University of Köln (D).]

4.5 THE REST OF THE WORLD

The portion of the industrial membrane industry outside of the U.S., Europe and Japan is negligible, except for a surprisingly vigorous program in Australia. There are three Australian-based membrane companies, Memtec, Syrinx and Aquapore. Of these, Memtec is the largest, with about 130 Australian employees and, since their acquisition of Brunswick Filtration Division in 1988, a substantial presence in the U.S. Memtec produces microfiltration equipment largely centered on water pollution control applications.

The Australian government is sponsoring membrane research at the level of about \$1 million annually.

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CHAPTER FIVE

ANALYSIS OF RESEARCH NEEDS

5.1 PRIORITY RESEARCH TOPICS

Based on group meetings and review discussions, a list of five to seven important research topics was selected for each of the seven major membrane technology areas. This list, totaling 38 research topics, was then ranked in order of priority. The list is shown in Table 5-1, together with the ranking scores assigned by each group member. A topic ranked number 1 received 38 points in the score column, a topic rated number 2 received 37, and so on. Since the review group had six members and there were 38 topics, the maximum possible score for any topic was 6x38 or 228.

A few points should be made about this priority list. First, although the research interests of the six author group members are completely different (this, in fact, was the basis for their selection), the priority rankings that they assigned were remarkably similar. Most of the group members had one or two topics, out of the 38, that they ranked particularly high or low compared to the average ranking. The deviations of the group member's individual rankings from the average ranking were, however, generally small. The standard deviation shown in the last column reflects the scatter between the individual group member's ranking of each topic. In general, the scatter was least at the top and bottom of the tables, reflecting good agreement between the group members on the most and least significant research topics. Not unexpectedly, there was most scatter in the middle range. Based on these scores, the top 10 priority research topics were selected. These topics are listed, with brief descriptive comments, in Table 5-2.

Table 5-1. Important Research Topics for the Seven Membrane Technology Areas, Ranked in Priority Order

RANK	TOPIC	A	B	C	D	E	F	Total Score	Std. Dev.
		Rank	Rank	Rank	Rank	Rank	Rank		
1	PV:Membranes for organic-organic separations	1	5	3	3	9	6	201	2.6
2	RO:Oxidation-resistant membrane	7	6	4	13	6	5	187	2.9
3	GS:Thin-skinned membranes	2	3	16	20	1	2	184	7.7
4	FT:Oxygen-selective solid carrier membranes	5	1	2	6	19	13	182	6.4
5	GS:High O ₂ /N ₂ selectivity polymer	6	4	1	2	25	10	180	8.1
6	UF:Fouling-resistant membranes	4	7	11	19	5	3	179	5.5
7	PV:Solvent-resistant modules	3	16	8	10	13	11	167	4.1
8	GS:Thin composite membranes	16	11	17	17	2	1	164	6.8
9	MF:Low-cost membrane modules	12	14	12	28	7	4	151	7.6
10	MF:Hi-T, solvent-resistant membranes & modules	19	9	20	5	4	20	151	7.0
11	ED:Temperature-stable membranes	15	18	15	18	17	7	138	3.8
12	GS:Membrane material for acid gas separation	8	30	27	1	3	22	137	11.6
13	UF:Lower-cost, longer-life membranes	13	12	7	29	16	16	135	6.8
14	UF:Low-energy module designs	26	35	5	14	8	8	132	10.9
15	PV:Membranes for organic solvents from water	9	19	21	9	15	26	129	6.2
16	RO:Improved pretreatment	14	21	10	31	21	9	122	7.6
17	PV:Membranes for dehydration of acids & bases	20	25	13	4	27	19	120	7.7
18	RO:Bacterial attachment to membrane surfaces	22	15	28	23	11	17	112	5.6
19	ED:Spacer design for better flow distribution	30	13	9	35	18	12	111	9.7
20	UF:Hi-T, solvent-resistant membranes & modules	21	36	14	8	22	25	102	8.8
21	RO:Increased water flux	36	17	23	12	10	31	99	9.5
22	MF:Non-fouling, cleanable, long-life membranes	11	10	24	30	33	29	91	9.1
23	FT:Olefin-selective solid carrier membranes	17	23	18	25	28	27	90	4.2
24	GS:Reactive treatments	10	31	33	16	31	21	86	8.6
25	GS:Oxygen-selective membrane	29	22	38	7	32	14	86	10.6
26	ED:Better bipolar membranes	23	2	36	22	37	24	84	11.6
27	GS:Selection methodology for separation matls.	24	27	34	15	20	28	80	6.0
28	UF:Hi-T, high-pH and oxidant resistant membranes	28	24	19	27	23	30	77	3.6
29	ED:Steam-sterilizable membranes	37	8	22	37	36	18	70	11.1
30	FT:Optimal design of membrane contactors	38	29	6	38	12	36	69	12.9
31	RO:Cleaning improvements	25	33	31	34	14	23	68	6.9
32	FT:Membrane contactors for copper & uranium	27	26	25	21	30	37	62	5.0
33	PV:Plant designs and studies	18	34	35	24	26	33	58	6.2
34	MF:Continuous Integrity testing	35	38	29	32	24	15	55	7.6
35	FT:Membrane contactors for flue gases & aeration	34	28	37	11	29	38	51	9.1
36	ED:Fouling-resistant membranes	31	20	32	36	38	32	39	5.7
37	MF:Cheap, fouling-resistant module designs	32	37	26	26	35	34	38	4.3
38	RO:Disinfectants	33	32	30	33	34	35	31	1.6

Table 5-2. Priority Research Topics in Membrane Separation Systems

Rank	Research Topic	Comments	Score
1	Pervaporation membranes for organic-organic separations	If sufficiently selective membranes could be made, pervaporation could replace distillation in many separations	201
2	Reverse Osmosis oxidation-resistant membrane	Commercial polyamide reverse osmosis membranes rapidly deteriorate in the presence of oxidizing agents such as chlorine, hydrogen peroxide, etc. This deficiency has slowed the acceptance of the process in some areas.	187
3	Gas Separation development of generally applicable method for producing membranes with <500Å skins	Would allow broad usage of advanced materials - even better if done in hollow fibers	184
4	Facilitated Transport oxygen-selective solid facilitated transport membranes	Air separations of higher selectivity are a target common to all types of membranes	182
5	Gas Separation higher O ₂ /N ₂ selectivity productivity polymer	Selectivity of 8-10 and permeability of 10 Barrer is required. Experimental materials approach these, but no ability to spin form them in hollow-fiber form has been reported. Most valuable as hollow fibers.	180
6	Ultrafiltration fouling-resistant membranes	Fouling is a ubiquitous problem in UF. Its elimination would boost total throughput >30% and reduce capital costs by 15% on top of eliminating cleaning. Better fractionation would also result, expanding UF use significantly.	179
7	Pervaporation better solvent-resistant modules	Current modules cannot be used with organic solvents and are also very expensive	167

Table 5-2. continued

Rank	Research Topic	Comments	Score
8	Gas Separation development of a generally applicable method for forming composite hollow fibers with <500Å skins	Only small amounts of the valuable selective material are required	164
9	Microfiltration high-temperature, solvent-resistant membranes and modules	Opportunity for ceramic or inorganic membranes. Potential uses include removal of particulates from coal liquids and replacement of bag houses in flue gas treatment	151
10	Microfiltration low-cost membrane modules	Huge potential applications will require commodity pricing, far from today's reality.	151

The highest ranked research topic was pervaporation membranes for organic-organic separations. A closely related topic, solvent-resistant pervaporation modules, ranked seventh in the priority list. The very high ranking of these two pervaporation research topics reflects the promise of this rapidly developing technology. The separation of organic mixtures by distillation consumes two quads of energy in the U.S. annually.¹ In principle, pervaporation could be used to supplement many existing distillation operations, for example by treating the top or bottom fractions from the distillation column. In some applications, such as ethanol/water separation or separation of organic/organic mixtures that form azeotropes at certain concentrations, pervaporation might displace distillation if appropriate membranes and equipment were available. If even a conservative 10% of the present energy expenditure on distillation were saved, this would represent 0.2 quads annually, or 10^5 barrels of oil daily.

The principal problem hindering the development of commercial pervaporation systems is the lack of membranes and modules able to withstand solvents at the elevated temperatures required for pervaporation. These problems can be solved. The development of membrane modules for a few special applications, for example,

the removal of methanol from isobutene methyltertbutyl ether (MTBE) mixtures, is already at the pilot-plant stage. Research breakthroughs in pervaporation appear imminent; widespread applications of the process could occur within the next decade if adequate research support were available. The impact on the nation's energy usage by the year 2010 could be substantial.

The second priority topic is the development of oxidation-resistant reverse osmosis membranes. The current generation of polyamide, high-performance reverse osmosis membranes have salt rejections of greater than 99.5% and fluxes three to five times higher than the cellulose acetate membranes developed in the 1970s. However, these membranes have not displaced cellulose acetate membranes because of their susceptibility to degradation by oxidizing agents such as chlorine, hydrogen peroxide or ozone. These oxidants are used to sterilize the membrane system. Periodic sterilization with high concentrations of chlorine is a requirement in food applications; low levels of chlorine are added to the feedwater of other reverse osmosis plants to prevent bacterial growth fouling the membrane surface. Methods of reducing the exposure of the membrane to chlorine have been developed, but these methods have reliability and cost problems. A number of groups are trying to solve the membrane degradation problem by modifying the chemistry of the polymer membrane. Progress has been made over the past 10 years, but membrane chlorine sensitivity remains a largely unsolved problem. The industry is also moving away from chlorine sterilization to ozonation. This emphasizes the need for a membrane with broad spectrum oxidation resistance rather than just chlorine resistance. If high-performance oxidation-resistant membranes were available, they could displace cellulose acetate membranes industry-wide, and a number of new applications for membranes would open up.

Development of ultrathin-skinned, gas separation membranes was ranked third in the priority research list; development of ultrathin, composite membranes was ranked eighth. The selection of these two closely related topics in the top 10 priority research list reflects the major impact that the development of generally applicable methods of making ultrathin membranes would have on the gas-separation industry. Development of this technology would also be of value in other membrane areas, particularly pervaporation.

The ultrathin-skinned, gas-separation membrane topic ranked number three refers to asymmetric membranes composed of one material. This would include membranes made by the phase inversion process, for example. The ultrathin composite membrane topic ranked number eight refers to multilayer membranes, in which a high-flux support is overcoated by an ultrathin permselective layer. Membranes of both types, made from a variety of polymers, by a variety of different techniques, are currently in commercial use. Asymmetric and composite membranes can be produced with a skin or permselective layer thickness down to about 0.5-1 μm . Membranes with permselective layers with thickness in the range of 0.1-0.5 μm are also made commercially, but the number of materials that can be formed into membranes of this type is very limited. Finally, there are a few claims in the literature of defect-free membranes being made in the range of 0.05 μm (500 \AA) or less. These claims must be treated with caution and it is certain that no generally applicable technique exists for forming this type of membrane.

New polymer materials are now being developed that do not lend themselves to fabrication into membranes by either the phase-inversion or the solution-coating technique, especially when very thin, <500 \AA , permselective layers are required. The development of membrane preparation methods, either for integral-skinned, asymmetric membranes or for multilayer composite membranes, that could be used to fabricate ultrathin membranes from any polymer material would therefore have a major effect on the entire gas-separation industry.

The energy impact of improved gas-separation technology is likely to be substantial. For example, if improved membranes for making oxygen-enriched air were available, it has been estimated that up to 0.36 quads of energy per year could be saved.² Removal of acid gases from sour natural gas could result in an energy savings of 0.01 quads per year in the processing of the gas alone.³ If the process enables the processing of very sour natural gas reserves that could not be exploited by other means, then the energy savings would be very large.

The development of facilitated-transport, oxygen-selective solid carrier membranes was given a research ranking of four. Liquid, oxygen-selective facilitated-transport membranes have been an area of research since the 1960s and some high-performance membranes have been produced in the laboratory. For

example, liquid carrier-containing membranes have been reported with oxygen/nitrogen selectivities of 20, compared to selectivities of 6 for the best commercial polymeric membranes.⁴ The permeability of the liquid membranes is also very high. Unfortunately, these liquid membranes are too unstable to be used in any commercial process. This instability problem has not been solved despite 20 years of research.

Recently, workers in Japan and West Germany have developed facilitated transport membranes using solid carriers.^{5,6} In these membranes the carrier is either physically dispersed in a polymer matrix or covalently bonded to the polymeric backbone of the matrix material. Contrary to accepted wisdom, these membranes exhibit substantial facilitation of the permeating species. The long-term stability of the membranes has not been demonstrated, nor have they been formed into high-performance, ultrathin membranes, but the solid carrier approach has merit. Although producing stable facilitated-transport membranes appears to be a high-risk research topic, the reward if this membrane can be made is correspondingly large. Stable membranes with an oxygen/nitrogen selectivity of 20, for example, would probably displace cryogenic processes as the production method for oxygen and nitrogen. Since nitrogen and oxygen are the first and third most important industrial chemicals in the United States, this would be a breakthrough of tremendous significance. Even more importantly, with these membranes it would become possible to produce oxygen-enriched air containing 40-80% oxygen at low cost. Availability of this oxygen-enriched air would dramatically alter the economics of many combustion processes. Although topic four is centered on the production of oxygen-selective carriers, it is likely the same technology, if successfully developed, could be applied to other separations, for example, the separation of acid gases from methane or alkane-alkene separations.

The production of highly oxygen-selective polymers was given a research priority ranking of five. The objective of this research topic is similar to topic four above. The target is, however, a good deal more modest and the prospects for success higher. The best commercially available oxygen-selective membranes have an oxygen/nitrogen selectivity in the range 6-7 and permeabilities of 2-10 Barrer. Systems based on these membranes are competitive for the production of

95-98% nitrogen on a small scale, up to 20-50 tons/day. They are not competitive for larger plants, where the economics of cryogenic separation are more favorable. Even small incremental improvements in membrane performance could, however, substantially increase the market share of membrane processes. If slight improvements in membrane performance were achieved, such that an oxygen/nitrogen selectivity of 7-10, with a permeability of 10 Barrer or more were possible, commercial production of oxygen-enriched air by membrane systems would become viable.

Reaching an oxygen/nitrogen selectivity target of 7-10 and a permeability target of 10 Barrer or more appears to be within sight. A number of materials with properties close to these values have already been reported. If they can be fabricated into high-performance membranes and modules, they could have a significant impact on the energy used in gas separation technology.

The principal problem in ultrafiltration technology is membrane fouling. For this reason, the development of fouling-resistant ultrafiltration membranes was given a research priority ranking of six. Fouling in ultrafiltration generally occurs when materials dissolved or suspended in the feed solution are brought in contact with, and precipitate on, the membrane surface. The precipitated material forms a secondary barrier to flow through the membrane and drastically lowers the flux through the membrane. The fouling layer becomes more dense with time, rapidly at first and then more slowly. The flux through the membrane declines correspondingly.

Fouling is usually controlled by rapid circulation of the feed solution across the membrane surface. The turbulence this produces in the feed solution slows the deposition of material on the membrane. Rapid feed circulation uses large amounts of energy, however, so a balance is struck between energy consumption and the amount of acceptable fouling. Fouling eventually reaches a point where even rapid feed solution circulation no longer maintains the flux at an acceptable level. The ultrafiltration system is then taken out of service and cleaned. Cleaning, however, almost never restores the system to its original performance and after some time, varying from 9 months to 5 years, the ultrafiltration modules must be replaced.

The development of fouling-resistant ultrafiltration membranes would decrease capital and operating costs and increase membrane lifetime. This is a difficult problem and no one solution is likely to be generally applicable. The basic mechanics of membrane fouling are undefined, so basic, as well as engineering, research is required. Promising approaches under development include modifying the membrane surface by making it more hydrophilic or adding charged groups to the surface. Membrane pretreatment with additives that coat the membrane to inhibit fouling is also used.

Solvent-resistant pervaporation modules, the seventh priority research topic, was discussed in conjunction with solvent-resistant pervaporation membranes (topic number one) and thin, composite gas separation membranes, the eighth priority research topic, was discussed in conjunction with thin-skinned, gas-separation membranes (topic number three).

Priority research topics nine and ten cover two research opportunities in the microfiltration area, namely, development of low-cost microfiltration modules and development of high-temperature, solvent-resistant membranes and modules. Development of low-cost modules was selected as a priority topic because a number of extremely large potential applications exist for microfiltration if costs can be reduced. These applications include numerous possibilities in water-pollution control applications. For microfiltration to move from its current role as an effective but relatively expensive technology, microfiltration modules will be need to be produced as a commodity with drastically lower costs. The authors believe this goal is desirable and achievable.

The development of high-temperature and solvent-resistant membranes and modules, the tenth priority research topic, would allow microfiltration to be used in a number of applications where the limitations of current membrane modules are a problem. These applications include filtration of hot wash-waters for recycling, filtration of refinery oils, and removal of particulates from various hot fluid streams. Ceramic membranes, which could be used in this type of application, are just entering the market. These first generation ceramic membranes are far too costly to be widely used, but as development efforts

progress, lower cost, more efficient ceramic filters may become available. Ultrahigh temperature performance polymers could also be used in this type of application.

5.2 RESEARCH TOPICS BY TECHNOLOGY AREA

In the preceding section, the 38 priority research topics were addressed by rank order. The same topics arranged by technology areas are listed in Tables 5-3 to 5-9. These tables were produced after several revisions suggested at the group meetings and by the external reviewers. Each table lists the top five to seven priority research topics in its area. A brief description of the research topic and the priority ranking is given, together with the prospect for realization of each particular topic. Topics with relatively low prospects for commercial success within 10-20 years were given a fair ranking in terms of prospects for realization. Topics where the prospects were considered better, but where the technology is still very undeveloped, or where major problems exist, were ranked good. Topics with a relatively high probability for successful commercialization, with minor problems, were ranked very good. Topics marked excellent were considered very highly likely to succeed within the next ten years with adequate research support.

Following each table is a summary of the relative merits and importance of the various items. A detailed discussion of the individual topic areas is given in the appropriate chapters in Volume 2.

5.2.1 Pervaporation

Table 5-3. Priority Research Topics in Pervaporation

Research Topic	Prospect for Realization	Comments	Rank out of 38
Membranes for organic-organic separations	Very Good	If sufficiently selective membranes could be made, pervaporation could replace distillation in many separations.	1
Better solvent-resistant modules	Excellent	Current modules cannot be used with organic solvents and are also very expensive	7
Better membranes for the removal of organic solvents from water	Very Good	More solvent selective membranes are required, especially for hydrophilic solvents (phenols, acetic acid, methanol, ethanol, etc.)	15
Dehydration membranes for acidic, basic, and concentrated aqueous solvent streams	Good	Would be of use in breaking many common aqueous-organic azeotropes.	17
Plant designs and studies	Good	Pervaporation will probably be used in hybrid systems for organic-organic separations. System design studies are needed to guide research.	33

Four of the five pervaporation research topics listed in Table 5-3 were ranked in the top half of the priority research list. Two topics relating to the development of pervaporation membranes and modules for the separation of organic mixtures were ranked in the top ten. This high ranking reflects the potential pervaporation has to replace or augment distillation in a number of significant applications in the chemical processing industry. Distillation is an energy-intensive operation that consumes 28% of the energy used in all U.S. chemical plants and petroleum refineries.⁷ The total annual distillation energy consumption is approximately 2 quads, or 3% of the entire national energy usage.¹ The top 10 distillation separations (Crude oil; Intermediate hydrocarbon liquids; Light hydrocarbons; Vacuum oil; Sour water; Ammonia/water; Styrene/Ethyl-benzene;

Ethylene glycol/water; Methanol/water; Oxygen/nitrogen) together consumed 1.0 quads of energy in 1981. Many of the major distillation separations consume more than 2,000 Btu/lb of product.

Pervaporation systems are currently used for breaking the water-alcohol azeotrope in the preparation of anhydrous alcohol. Process experience indicates that the steam requirement of pervaporation is 20% of that for azeotropic distillation for the preparation of 99.7% isopropanol from a feed stream containing 87% isopropanol.⁸ Pervaporation does have other energy requirements, which include electrical energy for vacuum pumps and chillers. However pervaporation still offers a 60% energy savings over azeotropic distillation in the dehydration of ethanol.⁹ It is likely that similar savings could be achieved in other separations where azeotropes are involved. Pervaporation could also be used to supplement many existing distillation operations, for example by treating the top or bottom fractions from the distillation column. A 10% reduction in energy consumption for distillation would save 0.2 quads of energy per year.

The other two pervaporation topics ranking in the top half of the priority list both relate to removal of solvents from aqueous streams. This type of stream is very common and pervaporation systems could be widely used in solvent-recovery and pollution-control situations. However, current membranes are best suited to recovery of relatively hydrophobic solvents. Developments of membranes able to treat more hydrophilic polar solvents and acidic or basic solvent streams would allow the process to be much more widely used.

5.2.2 Gas Separation

Table 5-4. Priority Research Topics in Gas Separation

Research Topic	Prospect for Realization	Comments	Rank out of 38
Development of generally applicable method for producing membranes with <500Å skins	Very Good	Would allow broad usage of advanced materials - even better if done in hollow fibers	3
Higher O ₂ /N ₂ selectivity ($\alpha \approx 7-10$) and productivity polymer (P $\approx 2-3$ Barrer for O ₂)	Very Good	Experimental materials approach these intrinsic α and P numbers, but no ability to spin form them in hollow fiber form has been reported. Most valuable as hollow fibers.	5
Development of a generally applicable method for forming composite membranes with <500Å skins	Good	Only small amounts of the valuable selective material are required.	8
Membrane material with high selectivity CO ₂ and H ₂ S separations from CH ₄ ($\alpha > 45$) and H ₂ ($\alpha > 20$) at high CO ₂ and H ₂ S partial pressures	Good	Will become more important as the acid gas partial pressure in the feed from EOR projects increases.	12
Reactive treatments for increasing the selectivity of a preformed ultrathin skin without excessive flux losses	Good	Attractive if it is generally applicable. Both photochemical and fluorination processes have been demonstrated on dense films and on a relatively thick (1 μ m) composite membrane, but not on thin (<1,000Å) membranes.	24
High oxygen selective membrane ($\alpha \approx 12-15$ for O ₂ /N ₂) with good stability and an O ₂ flux 0.5- 1 $\times 10^{-4}$ cm ³ (STP)/cm ² ·s·cmHg	Fair	Carbon fiber, inorganic or facilitated transport membranes may meet α and flux goals.	25
Guidelines to streamline selection of polymers for high efficiency separations	Good	Much progress has been made, but steady, long-term building of this capability provides a good basis for opening potential new markets and preventing displacement by foreign products.	27

Gas separation was considered to be a high priority area for membrane research. Three of the seven gas separation topics in Table 5-7 were listed among the top ten priority research topics. Gas separation research topics are divided into two areas; the first dealing with methods of making better, high-performance membranes, and the second dealing with development of membrane materials with improved selectivity and permeability.

Topics covering methods of making high-performance gas separation membranes were ranked 3, 8 and 24 in the priority research list. To fully exploit the potential of gas separation materials now available, membranes that are essentially defect-free and have permselective layers on the order of 500Å thick or less must be mass produced. Techniques have been developed that come close to this target with a few materials. However, generally applicable techniques are not available. A number of approaches are being explored and the prospects of success are good to very good.

The second major area of current gas separation research is the development of better membrane materials. In the past, membranes were prepared from polymers developed for other uses. The new generation of gas separation membranes just now entering the market all use membranes made from polymers specially designed and synthesized for their permeability properties. This area of research will continue to grow. Particularly important target applications are the separation of oxygen and nitrogen from air and the separation of acid gases, such as carbon dioxide and hydrogen, from natural-gas and chemical-process industry streams. Development of these new membrane materials has been aided by basic ongoing research aimed at understanding the effects of polymer membrane structure on permeability.

Estimates for the energy savings from oxygen-selective membranes vary widely, depending on the oxygen enrichment possible. Low grade oxygen enrichment (35%-50%) has been shown to be sufficient to improve the energy-efficiency of combustion processes. However if high grade (>75%) oxygen-enriched streams were available at low cost, then the process modifications and resultant

energy savings would occur throughout industry. Various estimates have placed the energy savings from the production of oxygen-enriched air at between 0.06 and 0.36 quads per year.²

Upgrading of 200,000 SCFD of sour natural gas (17% H₂S, 45% CO₂) to remove 30% of the acid gas present using a membrane system will result in an estimated savings of 0.01 quads per year.³ The total energy savings will depend on the economic feasibility of producing gas from sour gas wells and are potentially huge.¹⁰

5.2.3 Facilitated Transport

Table 5-5. Priority Research Topics in Facilitated Transport

Research Topic	Prospect for Realization	Comments	Rank out of 38
Oxygen-selective solid facilitated transport membranes	Fair	Air separations of higher selectivity are a target common to all types of membranes	4
Olefin-selective solid facilitated transport membranes	Fair	Membrane life is the key question especially with sulfide contaminants.	23
Optimal design of membrane contactors	Excellent	As membranes get better, module design maximizing mass transfer per dollar becomes key.	30
Membrane contactors for copper and uranium	Excellent	Dramatic success for drugs can be repeated with metals.	32
Membrane contactors for flue gas and aeration	Good	Success in the field is uncertain.	35

The five facilitated transport research topics are divided into two groups: research on oxygen-selective solid facilitated transport membranes, which was ranked very high, and all the other topics, which were ranked relatively low. Separation of oxygen and nitrogen from air continues to interest membrane research groups around the world. Facilitated transport membranes have been made in the laboratory with selectivities for oxygen from nitrogen of 20 or more.⁴ If this selectivity could be achieved in a stable industrial membrane it

would be a major breakthrough with an enormous economic impact. In principal, this membrane would allow oxygen-enriched air to be used in a large number of combustion processes to produce the same amount of useful energy, but use significantly less fuel. Having said this, the production of these membranes is likely to prove extremely difficult, although recent work by the Japanese has been encouraging.

The four other facilitated-transport membrane topics were ranked low because generally the applications did not seem large, were too far in the future, or did not appear to offer a major advantage over competing technologies.

5.2.4 Reverse Osmosis

Table 5-6. Priority Research Topics in Reverse Osmosis

Research Topic	Prospect for Realization	Comments	Rank out of 38
Oxidation-resistant membrane	Good	Commercial polyamide reverse osmosis membranes rapidly deteriorate in the presence of oxidizing agents such as chlorine, hydrogen peroxide, etc. This deficiency has slowed the acceptance of the process in some areas.	2
Improved pretreatment	Good	Improvement of classical pretreatment methods that will enhance the reduction of suspended solids in feed streams to reverse osmosis systems is desired.	16
Bacterial attachment to membrane surfaces	Excellent	Bacterial fouling of membrane surfaces reduces productivity. Affinity of micro-organisms for different membranes is markedly different. Elucidation of attachment mechanism is required to select optimal membrane material and surface morphology.	18
Increased water flux	Excellent	Commercial thin-film composite membranes operate at 30% of theoretical efficiency because of flow restrictions within the membrane. Modest improvement could reduce the energy consumption of the reverse osmosis process significantly.	21
Cleaning improvements	Excellent	Membrane cleaning is not always successful; it remains a trial and error operation.	31
Disinfectants	Good	Disinfectants that do not produce trihalomethanes are needed to control membrane fouling by microorganisms.	38

Five of the six reverse osmosis priority research topics related to problems associated with membrane-fouling and addressed various ways of tackling this problem. For example, chlorination of reverse osmosis feed waters is now required to prevent bacterial fouling of the membranes. However, chlorine

degrades interfacial composites, the best membranes currently available. Development of an interfacial composite membrane resistant to not just chlorine, but other oxidants, such as ozone or hydrogen peroxide, was ranked very high. Improved methods of pretreating the feed or preventing bacterial attachment to the membrane in the first place also ranked in the top half of the priority research list. Finally, better membrane cleaning methods and a search for alternatives to chlorine as a disinfectant were included on the list, although ranked of lesser importance.

The focus on the operating problem of membrane fouling reflects the importance of this problem to the reverse osmosis industry. It also reflects the very high performance of current membranes. The best membranes available have salt (NaCl) rejections of greater than 99.5% with corresponding water fluxes of $0.5 \text{ m}^3/\text{m}^2 \text{ day}$. The development of membranes with better salt rejections and/or higher fluxes would enable reverse osmosis operations to operate at lower pressures, but the impact on costs would not be dramatic. For this reason, development of higher flux reverse osmosis membranes was included as a research topic, but ranked in the lower half of the list.

5.2.5 Microfiltration

Table 5-7. Priority Research Topics in Microfiltration

Research Topic	Prospect for Realization	Comments	Rank out of 38
Low-cost membrane modules	Excellent	Huge potential applications will require commodity pricing, far from today's reality.	9
High-temperature, solvent resistant membranes and modules	Good	Opportunity for ceramic or inorganic membranes. Potential uses include removal of particulates from coal and oil liquids and replacement for bag houses in flue gas treatment	10
Non-fouling, cleanable, long-life membranes	Good	Critical for abattoirs, dairies, breweries and wineries. Must be tolerant of the industry-approved sanitizer.	22
Continuous integrity testing	Good	Applications where biological integrity is required need evidence of continued compliance, especially for remote and automatic operation.	34
Cheap, fouling-resistant module designs	Fair	Current modules foul rapidly, especially with solutions having high loadings of particulates. Better module designs are required.	37

Microfiltration is a well-developed membrane process. Commercially, it is the largest and most developed of any studied. It has a high rate of investment and a high level of success. The profitable products developed by this industry concentrate on high value applications such as pharmaceuticals, foods, chemicals for making semiconductor integrated circuits, etc. These applications are exacting, demanding and do not require commodity pricing. There are important applications at the mass usage end of the spectrum; perhaps even potable water and sewage treatment. These applications require a different sort of thinking about product design, manufacturing and pricing.

The other research topics on the microfiltration list were aimed at developing specific membrane modules that could expand the applications of microfiltration. Development of high-temperature and solvent-resistant membranes was considered to be a high-priority topic because it could open up significant markets for microfiltration in the petrochemical industry and in the filtration of hot gas streams. Similarly, development of a method of continuously monitoring the integrity of membranes would allow increased market penetration of microfiltration into the cold sterilization of foods, beverages and pharmaceutical products.

5.2.6 Ultrafiltration

Table 5-8. Priority Research Topics in Ultrafiltration

Research Topic	Prospect for Realization	Comments	Rank out of 38
Fouling-resistant membranes	Good	Fouling is ubiquitous in UF. Its elimination would boost total throughput >30% and reduce capital costs by 15% on top of eliminating cleaning. Better fractionation would also result, expanding UF use significantly.	6
Lower-cost, longer-life modules	Excellent	Lower cost modules with better fouling control are required.	13
Low-energy module designs	Excellent	Current module designs use large amounts of energy in feed recirculation to control concentration polarization and fouling. More efficient module designs would use less energy.	14
Solvent-resistant membranes and modules	Fair	Petroleum applications of ultrafiltration could be large. Will require high temperature, solvent resistant membranes and modules. Ceramic membranes would fit here.	20
High-temperature, high-pH and oxidant-resistant membranes	Good	Current membranes cannot treat important industrial streams because of temperature, pH and oxidant sensitivity; another potential application for ceramic membranes.	28

Of the developed membrane processes, ultrafiltration was ranked highest as an area for increased research attention. This reflected the opportunities for further growth of this technology if unsolved problems are addressed. The biggest ultrafiltration research problem is membrane fouling; three of the five ultrafiltration research topics, ranked 6, 13 and 14, addressed various aspects of this problem. Fouling-resistant membranes is clearly a preferred research topic, but improved modules which are lower in cost and inherently more fouling-resistant, or modules which use less energy to control fouling, were other approaches given high priority.

Finally, the development of membranes and modules able to treat solutions at high temperatures, at high and low pHs, and containing solvents was considered to be a significant opportunity for ultrafiltration research, but of less importance than fouling-control research. Current membranes and modules are almost all polymer based and cannot be exposed to harsh environments. Ceramic membranes are being developed that have promise and are finding niche applications. If the cost and reliability of these modules could be improved, a number of significant opportunities for large-scale use of ultrafiltration would develop.

Both ultrafiltration and microfiltration could find new or broader applications in the food industry with attendant energy savings. The food industry uses 1.5 quads of energy per year.¹¹ Areas where the use of membranes could result in energy savings include:

- Concentration of corn steepwater and potato byproduct water
- Degumming, refining and bleaching of edible oils
- Clarification and concentration of beet sugar juice
- Bioprocessing of potato and dairy wastes
- Solvent recovery in edible oil processing

The potential energy savings in these areas are estimated at 0.13 quads annually.¹¹

5.2.7 Electrodialysis

Table 5-9. Priority Research Topics in Electrodialysis

Research Topic	Prospect for Realization	Comments	Rank out of 38
Membranes with better temperature stability	Excellent	Current ED systems are limited by operating temperature. Temperature-resistant modules would lower the electrical resistance and reduce energy use.	11
Spacer design for better flow distribution	Good	Concentration polarization remains a problem in electrodialysis. Better spacers would help.	19
Better bipolar membranes	Very Good	Bipolar membranes could be a major growth area in electrodialysis if better membranes can be made.	23
Steam-sterilizable membranes	Very Good	Electrodialysis is making inroads into the food and drug industry, but sterilization remains a problem.	29
Fouling-resistant membranes	Very Good	Fouling remains a problem in some electrodialysis applications.	36

Electrodialysis is an established membrane separation process which has changed little in the last ten years. For this reason, the five priority research topics in the electrodialysis area all addressed specific engineering problems. The highest priority rankings in Table 5-9 are both aimed at improving the current major application of electrodialysis, namely desalination of brackish waters. Membranes with better temperature stability and spacers with improved flow distributions would produce incremental improvements in brackish water desalination systems. Almost a billion dollars worth of electrodialysis systems are installed worldwide. Consequently, an incremental reduction in operating cost, of as little as 10%, by retrofitting better membranes and spacers, would produce a substantial savings.

The remaining three priority electrodialysis research topics were aimed at making electrodialysis more useful for various niche applications. For example, the application of electrodialysis to the food and pharmaceutical industries would be helped by more fouling-resistant membranes and stream-sterilizable membranes. Better bipolar membranes would be useful in the production of low grade acid and alkali. All of these applications were ranked fairly low, principally because the importance of the particular applications they addressed was not large.

5.3 COMPARISON OF DIFFERENT TECHNOLOGY AREAS

As was shown clearly by Table 5-1, the relative importances of the research priorities in different technology areas were ranked very differently. For example, the highest priority topic in electrodialysis, temperature-stable membranes, ranked almost equal with the fourth highest priority topic in gas separation, membranes for acid-gas separations. All but the highest priority item in facilitated transport ranked about level with, or below, the lowest priority items in ultrafiltration or gas separation.

Averaged rankings of the topics in each technology area are given in Table 5-10.

Table 5-10. Overall Ranks of the Seven Membrane Technology Areas

Membrane Technology Area	Average Research Topic Priority Ranking
Pervaporation	14.6
Gas separation	14.9
Ultrafiltration	16.2
Reverse Osmosis	21.0
Microfiltration	22.4
Electrodialysis	24.2
Facilitated transport	24.8

Clearly, research in the general areas of pervaporation and gas separation was ranked substantially higher than the other technology areas. This high ranking reflects the general feeling of the group that these two technologies

offer the best opportunities for research breakthroughs that would have a major effect on energy consumption and costs in U.S. industry.

The three established membrane filtration processes, ultrafiltration, reverse osmosis and microfiltration were grouped together in the center of the list spanning the average ranking. As a group, the ultrafiltration-related topics were ranked most important, followed by reverse osmosis, then microfiltration. All three topics scored one entry in the top 10 rankings, and microfiltration scored two. The priority topics in each area were remarkable similar. All of the areas included priority research topics covering fouling-resistant membranes and modules, membranes and modules that can withstand harsh environments, and lower cost modules.

Module fouling is a continual problem in all membrane filtration processes, and the high priority given by the author group to ways of reducing fouling reflects the importance of the problem. Fouling-resistant membranes for ultrafiltration ranked seventh out of 38, improved pretreatment to reduce fouling and reduction of bacterial fouling, both for reverse osmosis, ranked sixteenth and eighteenth, and nonfouling microfiltration membranes ranked in position twenty-two. Methods of reducing the cost of modules and improving module design also ranked high.

Electrodialysis and facilitated transport were both marked at the bottom end of the research priority list about equal in level of importance. In the case of electrodialysis, the authors generally felt that electrodialysis is a well-developed process with a few established large applications. Electrodialysis does not appear to be as widely applicable to problem separations as other membrane technologies, such as reverse osmosis, ultrafiltration or microfiltration. For this reason it was ranked low. The low rank of facilitated transport reflected general disenchantment with the process. Liquid facilitated-transport membranes with very high selectivities and fluxes have been available for more than 20 years, but there are no commercial plants in operation. The problems of membrane and carrier instability have just proven too intractable.

5.4 GENERAL CONCLUSIONS

One of the primary goals of the U.S. Department of Energy is to foster and support the development of energy-efficient new technologies. The primary objective for energy-efficient technology is a strategic one: to reduce U.S. energy consumption, thereby reducing the oil trade deficit and the dependence on foreign sources of oil. The energy costs of an industrial process directly affect the cost of the goods produced. Therefore energy-efficient production technology can result in higher productivity gains, an increase in the international competitiveness of U.S. industry and a reduction of the current trade deficit. Processes that use energy inefficiently are also significant sources of environmental pollution. Environmental concerns have added impetus to the search for energy-efficient, environmentally safe technologies. One such technology is membrane separation, which offers significant reductions in energy consumption in comparison with conventional separation techniques.

Membrane separation processes are widely used in many major industries. Total sales of industrial membrane separation systems are more than \$1 billion annually.¹² The United States is the dominant supplier of these systems. United States dominance of the industry is being threatened, however, by Japanese and, to a lesser extent, European companies.

The focus of this project was to report to the U.S. Department of Energy on recommendations for priority research needs in membrane separation science and technology. These specific aspects are discussed in the previous sections. Set out here are some general conclusions relating to DOE's support of membrane research.

Conclusion 1. DOE and other Federal spending on membrane-related research is small and fragmented: Current total Federal support for membrane-related research is on the order \$10-11 million/year. Of this total, approximately \$4-5 million is provided by the National Science Foundation (NSF) to support basic membrane research, mostly in the universities. A further \$2-3 million is used by the Environmental Protection Agency (EPA), the Department of Defence (DOD),

and the National Aeronautics and Space Administration (NASA) to support various membrane activities that relate directly to their missions. The final \$4 million is used by the Department of Energy (DOE) to sponsor energy-related research programs. Various offices within the DOE support programs in their own particular area of interest. The Office of Industrial Programs funds research at about the \$1.5 million/year level; the Office of Basic Energy Research funds about \$1 million/year, and the Office of Fossil Energy about \$1-1.5 million/year.

In contrast, Federal research support was at a much higher level in the 1960s and 1970s. The lead agency was the Office of Saline Water (later the Office of Water Research and Technology), which sponsored \$20-40 million/year of membrane-related research activities for many years. This high-risk investment reaped handsome rewards, going far beyond the originally contemplated scope of the program and impacting several different areas of membrane technology, which are still being enjoyed by the U.S. economy.

Current U.S. Government membrane-related research programs, from all agencies together, are approximately half of the corresponding Japanese and European efforts. Other governments have attached greater importance to furthering the advance of membrane science and technology. Without increased commitment and support to membrane-related topics, the United States may begin to lose markets in the existing membrane technologies, and may be a junior player in world markets for the emerging membrane technologies.

Conclusion 2. Engineering problems are holding the U.S. membrane industry back: A noteworthy aspect of the research priority list was the heavy emphasis on membrane technology and engineering, rather than membrane science. Engineering- or technology-related problems ranked in positions 3, 5, 7, 8, 9 and 10 in the top 10 priority list. Other items that have an engineering component include development of high-performance oxygen/nitrogen separation membranes and modules, for which some suitable polymer materials are already known, but where the technology to form them and use them is lacking. Even an item such as the first-ranked priority topic, pervaporation membranes for organic/organic separations, which at the moment requires basic membrane development and testing studies, will not be able to be exploited industrially, with the attendant

major energy-savings benefits, unless the membrane development goes hand-in-hand with the ability to form modules and design systems able to handle the environment in which the pervaporation process is performed.

At present, a large portion of the total monies provided by Federal sources is devoted directly to basic scientific research programs. As is right and proper, essentially all of the \$4 million support for research from the NSF is devoted to fundamental membrane science. The projects funded by the DOD and NASA, together amounting to no more than about \$1 million annually, are a mix of basic and engineering items, but highly specialized and out of the mainstream of membrane development. EPA spends \$1.5 million/year, mostly on applications- and engineering-oriented programs. The DOE's \$4 million annual expenditure on membrane research is diverse. The Division of Chemical Sciences of the Office of Energy Research, for example, typically funds fundamental programs, whereas the other branches of DOE fund a spectrum of programs ranging from theoretical or modeling studies to heavy engineering. In total, it appears that, of the \$10-11 million available annually to membrane topics, less than \$4 million is probably spent on engineering-related projects.

The emphasis of the expert group on technology and engineering issues reflects the current developed status of the membrane industry. The state-of-the-art in the emerging, as well as the established technologies, shows that engineering issues are central to the ability to achieve practical, economically viable, energy-efficient membrane systems.

Conclusion 3. Key strategic focus areas are pervaporation and gas separation:
If pervaporation could displace or supplement distillation in sectors of chemical processing, the effects on energy consumption and competitiveness of U.S. industry would be substantial. At present, the United States trails third in the world in pervaporation research effort and capabilities. It is apparent that both the Europeans and the Japanese have recognized the important potential of the technology. In gas separation, where the United States is still first in the field, ground may be lost as other countries step up their efforts. A focused effort in gas separation technology is needed if the United States is to be a leader in the new generation technology. The attendant benefits would be that membrane-based

gas separation will become competitive with conventional, energy-costly separation technologies over a much broader spectrum.

Conclusion 4. Government support is important: Federal support remains crucial to the membrane industry, both developing and developed. In the United States, innovation typically comes from universities, small companies, or small groups within companies. This has been especially true in the membrane industry. The microfiltration industry, the area that currently commands more than half of the total revenues generated by membrane sales, has been built up by dedicated companies, a number of which, such as Gelman, Gore, Amicon and Pall, started literally as one-man bands. The same is true in reverse osmosis, where companies like Desalination Systems and Osmonics were built on the new technology. In both of these industries, early U.S. Government support was a key factor in future success. Membrane research is being conducted in a number of large companies, but in general the research effort is fragmented, and a sizeable portion of the R&D effort is coming from small innovators. It was felt that, in the emerging technologies in particular, the leadership, focusing and commitment roles played by Federal agencies in the past are still essential if progress across a broad front is to be stimulated and maintained.

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APPENDIX

APPENDIX A. PEER REVIEWERS COMMENTS

A draft final version of this report was sent to ten outside reviewers. The reviewers were chosen for their experience and background in membrane science and technology and their knowledge of the membrane industry. The following people served as peer reviewers of this report:

Dr. J. L. Anderson (Carnegie Mellon University)
Dr. J. Henis (Monsanto)
Dr. J. L. Humphrey (J. L. Humphrey and Associates)
Dr. S.-T. Hwang (University of Cincinnati)
Dr. N.N. Li (Allied Signal)
Dr. S. L. Matson (Sepracor, Inc.)
Dr. R. D. Noble (University of Colorado)
Dr. M. C. Porter (M. C. Porter and Associates)
Dr. D. L. Roberts (SRI International)
Dr. S. A. Stern (Syracuse University)

As far as possible, the reviewers' comments, particularly those dealing with specific changes or corrections, were incorporated directly into the report. Excerpts from the reviews, covering general comments, policy recommendations and dissenting views are presented in this section along with the authors' rebuttals.

A.1 GENERAL COMMENTS

Three features of the report drew comments from many reviewers. The first concerns the balance of the report between emphasis on basic science and emphasis on engineering issues. The second concerns the importance of integrating membrane technology into hybrid treatment systems. The last concerns the merits or demerits of the ranking scheme that was adopted by the group.

A.1.1. The report is biased toward engineering, or toward basic science.

Dr. Alex Stern commented that "the list of research priorities is too much skewed toward practical applications". Dr. Stern expressed concern at the "decline in long-range fundamental research in this country". His opinion was that "applied research and development can solve many operational problems and

improve the efficiency of existing membrane separation processes. However, only fundamental research can generate the new concepts which will produce the membrane processes of the future."

Dr. John Anderson pointed out that "the major emphasis of this report is on the research needs for membrane engineering and technology..... The panel were composed primarily of industrial researchers with a few academic persons scattered throughout. The science of membranes (how they work, structure versus function) was given low priority for this study".

Dr. Steve Matson also observed that "high priority is given in the study to engineering and product oriented research".

Dr. Jay Henis expressed a completely opposite view. Dr. Henis said that there was too much emphasis on basic research issues, and stressed that the research topics need a greater engineering emphasis. He believed that most of the top priority items have not adequately addressed engineering issues, and that, if engineering input had been included in the analysis, the priorities might have been different.

A.1.2 The importance of integrating membrane technology into total treatment systems.

Dr. Steve Matson said that "it is very difficult to dispute the essential conclusion of the study that pervaporation and membrane gas separations are two areas in which increased federal funding would likely have great and relatively near-term impact on energy consumption in the chemical process industry. This reviewer might have put hybrid membrane processes (not-just pervaporation-based) a bit higher on the priority list, for example, and he might have lobbied for more consideration of important problems in biotechnology that are addressable with membranes and which have important energy and environmental implications".

Dr. John Anderson stated that the "concept of systems design with membrane technology integrated into the design is ignored. No persons active in design research were on any of the panels. This omission significantly weakens the

statements made on behalf of the potential of membranes, for the real potential of membrane separations will only be achieved when they are formally integrated into process design methodology".

REBUTTAL: The expert group acknowledges that hybrid designs are very important. The advantages of combining distillation and membrane separation, for example, are discussed in Volume II, Chapter 2, Pervaporation. However, hybrid systems are only useful where the membrane process will complement an existing separation operation to provide technical or economic advantage. Such opportunities clearly exist for the emerging technologies of pervaporation and membrane gas separation, particularly in the process industries. The mature membrane technologies, however, tend to be stand-alone, for example desalination by reverse osmosis, and many microfiltration and ultrafiltration applications, or their potential for inclusion in an integrated separation process has already been recognized and is not likely to be substantially changed by improvements in the membrane process.

A.1.3 The ranking scheme.

Dr. John Anderson was bothered by the rankings. "Besides some possible vested interest by panel members", he believed that the rankings are "too loosely assigned and might lead to biased funding in one area at the expense of another equally important area. I strongly recommend that the top 10 or 15 areas be listed without a priority ranking" but rather "be viewed as a collection of equally important individual topics".

Dr. Richard Noble accepted the ranking scheme, but would have preferred that the ranked items be grouped together by according to theme. His point was that "there are common themes or research needs that "permeate" this field. Advances in a particular theme in one membrane area can have a synergistic effect in other areas." Dr. Noble advocated DOE support of the following general themes: Membranes with Improved Resistance, Membrane Fouling, Thinner Membranes, Membrane Materials and Use of Reaction Chemistry. He deprecated support of themes relating to Membrane Treatment, Modules, and Standards, Criteria and Testing.

Dr. Steve Matson preferred to rank the 38 items only in terms of high, medium or low priority. His high-priority items all fell within the top ten rankings, and his low-priority items all fell below ranking 17.

Drs. Sun-Tak Hwang and Mark Porter also provided their own rankings, both of which were in very good agreement with the consensus of the expert group. Dr. Hwang ranked most ultrafiltration topics a little higher than the report rankings; Dr. Porter ranked gas separation topics generally higher, and reverse osmosis and microfiltration topics generally lower than the report rankings.

REBUTTAL: The goal of the study, and, therefore, the objective of the group, was to prepare a prioritized list of research needs. All of the 38 topics considered were significant enough to enter the analysis. The rankings were prepared by secret vote of the group of authors, whose personal biases, if any, were mitigated by the rest of the group. While one may disagree with the concept of ranks, examination of the scores in Table 5.1 shows that there is a clear consensus on certain definite levels of priority that should be assigned.

A.1.4 Comparison with Japan

Two reviewers, Dr. Jay Henis and Dr. Richard Noble, drew comparisons between membrane technology in the United States and Japan. Dr. Noble urged that "Government funding of membrane-related research is important and essential". His view was that "DOE should facilitate partnerships and/or collaborative efforts between universities and industrial companies to make fundamental advances and rapidly transfer the knowledge to the private sector so it can be implemented and commercialized. This is the approach being taken in Japan and Europe and uses the talents and resources of everyone who can aid in advancing the knowledge base and implementing the knowledge".

Dr. Henis was concerned that the Japanese have been producing better products with our basic science. He felt that what the United States needs is a strongly practical approach. He stressed that good science should not be restricted to fundamental issues, but should also include engineering and applications considerations.

A.2 SPECIFIC COMMENTS ON APPLICATIONS

A.2.1 Pervaporation

Pervaporation research ranked number one in the priority chart and not surprisingly, therefore, attracted comments from all the reviewers.

Dr. Jimmy Humphrey called for more emphasis on hybrid applications. He said that high purity distillation requires high reflux ratios, which in turn increase the steam requirements. He pointed out that, for instance, pervaporation could be used in a hybrid arrangement to treat the overhead product from a distillation column to produce a high purity stream.

Dr. John Anderson said that the case for pervaporation is overstated, or at least not supported. His opinion was that recent advances in multicomponent distillation with respect to energy conservation and azeotrope breaking will reduce the impact of pervaporation. He believed that pervaporation will not replace distillation over the next 50 years, although it may prove valuable in supplementing distillation in the separation of organic liquids.

Dr. Richard Noble expressed the view that the development of solvent-resistant modules is not worthy of DOE support and is best left to funding by venture capital.

REBUTTAL: If pervaporation is to be used either as an alternative to distillation or to complement distillation, then both membranes and modules that can handle the environment in which organic/organic separations take place will be required. For DOE to support membrane development but not module development is inconsistent, and creates a risk of the membrane technology being either wasted or taken up and developed outside the United States. The effort supported by the Office of Saline Water to develop reverse osmosis technology embraced both membranes and modules, and proved very successful.

Dr. Jay Henis, like Dr. Humphrey, took the view that current distillation technology, with best available energy recovery systems, should be considered in evaluating the relative merits of pervaporation. He felt that new pervaporation

units would be used in the basic chemical industries, which are presently in decline in the United States and are increasingly located off-shore, so that the domestic energy savings resulting from pervaporation will not be large.

REBUTTAL: The report recognizes that hybrid systems may be where the real potential for certain pervaporation applications lies. For strategic and practical reasons, the United States will always have a large petrochemical industry, and this is an industry segment where pervaporation will both find applications and result in energy savings. Besides the basic chemical industries, pervaporation could be used in the chemical process industries, food processing, wastewater treatment and many other specific applications.

A.2.2 Gas Separation

Several reviewers made specific comments expressing their own ideas as to the most significant areas on which to focus. Dr. Richard Noble thought that the breakthrough will be in new materials, such as inorganic membranes, zeolites and molecular sieve membranes. He felt that most of the limitations of present gas separation technology arise from the polymeric membrane materials.

Dr. Alex Stern stressed the importance of fundamental research into molecular dynamics, which would lead to the ability to predict diffusion coefficients from basic physico-chemical properties, and the design and synthesis of new materials created exclusively for their permeation properties.

Dr. Jay Henis believed that the development of a membrane to remove carbon dioxide and hydrogen sulfide from low-grade natural gas, ranked 12 in the priority list, has been rated too low, and urged that such a membrane could have a measurable, instantaneous impact on U.S. energy reserves. Dr. Henis also questioned the importance of the development of ultrathin-skinned membranes. His view was that the problem of membrane productivity could be addressed by other means, such as increasing the free volume of the polymer.

A.2.3 Facilitated Transport

Most reviewers concurred with expert group opinion that the general prospects for facilitated transport are not bright. However, Dr. John Anderson

believed "a major breakthrough is possible with facilitated transport; however, new research concepts are needed here. Thus, I would argue that with respect to this topic, membrane science should be supported by DOE, and the science should be truly novel (i.e., not just another species of mobile carrier in a liquid film)". Dr. Richard Noble thought that there will be niche applications for facilitated transport in 10 years time, and he would have liked to see oxygen/nitrogen selective facilitated transport membranes included in the discussion of gas separation membranes.

Dr. Jay Henis said that facilitated transport deserves a very low or zero priority, because the combination of requirements is impossible for a real system. He pointed out that solid carriers are active species, not unlike catalyst molecules, and are subject to the same poisoning processes, and that liquid membranes require an infinite partition coefficient for the carrier between the membrane and the process streams to prevent the carrier from being leached out.

A.2.4 Reverse Osmosis

Dr. Jay Henis wanted clarification that oxidation-resistant membranes, ranked 2 in the priority list, should cover membranes that will resist oxidants other than chlorine. He stated that the industry trend is toward ozonation, and that membrane research should, therefore, be directed at membranes that could withstand various oxidants.

Dr. Noble felt that most of the research needs identified for reverse osmosis were more appropriately within the province of the Department of the Interior, and should not be funded by DOE.

A.2.5 Ultrafiltration

Dr. John Anderson commented that "work on fouling-resistant membranes is certainly needed, but the scope of this research should include development of easily cleanable and restorable ultrafiltration membranes. These might not be polymer-based."

Dr. Richard Noble thought that more research is needed on ceramic and inorganic membranes. "They can be cleaned, sterilized, and put in hostile

environments much more easily than polymer films. They are a high-cost item now but research will inevitably lead to lower costs and materials suited to various applications."

Dr. Alex Stern believed more fundamental research should be supported, such as using Monte Carlo techniques to calculate particle trajectories and predict gel layer buildup and fouling rate. He felt that these basic insights can contribute to more efficient membrane and module design and low-energy operation.

A.2.6 Microfiltration

Dr. Richard Noble was of the opinion that low-cost module development is best left to market forces and should not be supported by the DOE.

A.2.7 Electrodialysis

Dr. Richard Noble stressed that bipolar membranes and better module design are important.

A.2.8 Miscellaneous Comments

Dr. John Anderson and Dr. Steve Matson were both concerned about the scope of the study. Dr. Anderson said "The entire area of biochemical/biomedical membrane separations is omitted. This promises to be a big dollar item, and energy will certainly play some role here on products of modest volume. In my mind, it is not inconsistent for DOE to consider supporting research on large-scale bioseparations by membrane methods." Dr. Matson expressed himself "somewhat distressed by the scope of the present study: i.e., by what is not covered by the study as opposed to what is. Its limitation to relatively well-developed membrane technologies and industries is a very significant one, especially in the context of a "research needs" assessment. While the study sets out to consider four "fully-developed" membrane processes and two "developing" processes, it examined only one "to-be-developed" technology -- namely facilitated transport -- and that a technology which is over 20 years old. Thus, the study deals primarily with an assessment of the state of the art and with what can reasonably be expected to advance it." Dr. Matson suggested a follow-on study focused on "embryonic or emerging membrane technologies (e.g., the use of sorbent membranes in high-flux adsorption processes, the use of catalytically

active membranes in reaction processes, the exploitation of attributes of membranes other than the permselectivity, and the like." Dr Richard Noble also would have liked to see catalytic membrane reactors included in the study, and would have liked to see more discussion of the use of facilitated transport membranes in sensors.

An additional study was also an idea broached by Dr. Norman Li, who felt that "the discussions of the effect on environmental quality were diffused and not very clear. Since this is an important issue, perhaps a separate volume to discuss air and water purification via various types of membranes would be a more focused and useful approach."

Both Dr. Norman Li and Dr. Jimmy Humphrey asked for a detailed breakdown of NSF's programs in membrane research.