

Pressure-Driven Functional Polymeric Membrane Technology as Athermal Separation Unit Operation in Chemical Engineering : A Review

Aspiyanto* , Agustine Susilowati, Yati Maryati, Hakiki Melanie, Hani Mulyani, Setyani Budiari, Anasthasia Fitria Devi, Nina Artanti, Yulia Anita, Euis Filaila

Research Center for Chemistry-National Research and Innovation Agency (NRIA-BRIN), KST BJ Habibie, Build 452, Serpong-15314, South Tangerang, Banten, Indonesia.

***Corresponding Author**

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ABSTRACT

This manuscript provides an overview of published scientific and trade literatures and a collection of useful reference information on all aspects of membrane science and technology, selected result of experiment applications, and more recent developments in pressure-driven flat sheet membrane (PDFSM) processes as new frontier and athermal separation unit operation in chemical engineering covering general description of the basic principles of membrane separation processes, benefits and drawbacks, and future trends of membrane developments. Meanwhile, experiment results of selected applications of membranes are removing and/or reducing bacteria, and recovering high protein and low fat from skim milk by microfiltration (MF) membrane (Fluoro polymer, 0.45 µm, Alfa Laval) as an alternative to replace heat sterilization, separating and/or concentrating protease enzyme by ultrafiltration (UF) membrane (Polysulphone, 20000 MWCO, DSS), separating target and valuable components in corn steeping water by using nanofiltration (NF) membrane (Thin Film Composite on Polyester, DSS), and separating and/or purifiing ions component in brackish water and sea water by reverse osmosis (RO) membrane (Thin film composite on Polypropylene, DSS).

Keywords: Membrane, Pressure, Microfiltration, Ultrafiltration, Nanofiltration, Reverse Osmosis.

INTRODUCTION

Background

Now-a-days, human beings and survival facing global challenges, such as growing population, increasing demand for water, energy and food, limiting raw material (natural) resources, advancing technological sector, developing economic aspect, and protecting environmental have been forced to move forward to growth and development of sustainable society in earth planet **[1].** In recent years, to recovery desired and valuable components with high-quality products from potential and prospect natural sources, to produce portable water from brackish and sea water, and to concentrate, to purify or to fractionate macromolecular mixtures in the food and pharmaceutical products has attracted great attention due to their potential uses to supply communities and industry with high quality water, In order to solve this complex problems, separation process technology of functional polymer membrane is one of the most direct, effective and feasible approaches to replace conventional separation techniques **[2].** Family of pressure-driven membrane processes for various applications in both upstream and downstream technology has emerged via a viable breakthrough into an essential, versatile, greener environmentally-friendly and athermal separatiom unit operation in chemical engineering with full of future possibilities **[3].** It is used to separate, clarify, debacterize, fractionate, purify, or concentrate a micro-, fine particles and colloidal region/level to ions and low MW solutes in molecular region/level from suspensions to aqueous solutions in the food and beverage, milk and dairy, pharmaceutical and medicinal, biotechnology industries, and desalination and water treatment over the last decade **[4].** One of

the success of membrane-based process separation technologies has come to symbolize the growth of MF, UF, NF, and RO themselves **[5].**

Objective

This review paper will essentially discuss some of the pressure-driven membrane separation techniques, which has been employed in selected application. Beyond membranes themselves, this review covers the basic principles of membrane separation processes, benefits and drawbacks, and future perspective, snapshot and outlooks of membrane separation technology developments.

Basic Principle of Membrane Separation Process

The term membrane referred to the thin layer of selective semi-permeable material with the thickness of the top active layer being approximately less than 1.0 μm **[6].** A membrane separation phenomenon is based on the ability of specific thin semi-permeable membrane of the appropriate physical and chemical nature acting as a permselective barrier, regulating the rate of transport of solvents or molecular and ionic components across a membrane to discriminate physically between molecules on the basis of difference in size range related to its molecular weight cut-off (MWCO) and to a lesser extent on molecular shape, facilitating separation of the target components from a liquid solution, enriching certain components in a feed stream (retentate) and depleting it of others (permeate). It moves through the membrane faster than other components of a mixture controlled by an pressure driven as a driving force. **[7].** Various the driving force, such as pressures, concentration gradients and chemical or electrical potential differences, may be adopted for membrane separations. All the pressure driven-based membranes are similar to each other, the only differences are the degree of semipermeability and the separation characteristics. The MF and UF are considered as filtration methods with low operation pressure, whereas NF and RO are expressed as concentration method with relatively high operation pressure. Based on different criteria including effective pore size of the membrane structure, size ranges of certain common constituents removed, function of the membrane type, driving force, and mechanism of separation, membrane processes can be classified into different categories, as MF, UF, NF, and RO **[8].**

Separation of MF mainly occurs through sieving mechanism due to the relatively large pore diameter size (approximately 0.1 μ m – 1 μ m), and involves the use of low gradient of operation pressure (0.5 – 3 bar). It means that MF will be used for the specific separation of the particles or emulsified and suspended solids, bacteria, organic colloids, and yeast cells in liquids with sizes micrometers scale range of $0.1 - 10 \mu m$ or molecular weights of > 200 kDa. UF membranes also physically operate based on the principle of sizeexclusion and sieving mechanism. They have a wider separation ability range than MF and depending on the pore size (generally between $0.01 \mu m - 0.1 \mu m$) or MWCO ranges 1,000 Da. to 300,000 Da. and, can remove particles, pathogens, viruses,and colloids like proteins from small molecules. UF operates at lower pressure (2 – 10 bar) **[9].** The term nanofiltration (NF) is derived from the fact that this membrane pore size value correspond to hypothetical pores of approximately in the range of 0.5 nm to 2 nm with relative molecular weight cut-off (MWCO) as low as 200 Dalton (Da.) to as high as 1,000 Da. and it is used to separate various molecules and macromolecules with relative molecular masses ranging from 200 Da. to 1,000 Da. **[10].** NF operates generally at a Trans Membrane Pressure (TMP) between 5 and 40 bar **[11].** NF is the family of pressure-driven membrane processes, which is considered to operate in the realm with separation capabilities between UF and HF or RO, and exhibits features of both. If the solvent flow through the polymer matrix (selective active semi-permeable membrane) is by convective flow through pores, it is stated as ultrafiltration (UF), meanwhile if solvent flow through the polymer matrix (membrane) is by diffusive, it is expressed as reverse osmosis (RO) **[12].** Due to their structural features, nanofiltration membranes have a dense active layer that contains charged functional groups on its surface. As a consequence, NF membranes selectivity for multivalents ions are not based only on the sieve effect (size exclusion, size-selective, steric hindrance), but also on ion-selective due to electrical interactions between the charged (Donnan exclusion, dielectric exclusion) NF membrane and the ions in the aqueous solutions of monovalent anion salts, and some di- and multivalent anion salts and larger organic compounds with a molecular weight (MW) **[13].** To understand basic principle of the reverse osmosis (RO), it is necessary to know osmosis itself. Osmosis is a natural

phenomenon in which a pure solvent (usually water) molecules will flow through a very dense semi-permeable membrane from the solution of the low concentrated solute side into the solution of the higher concentrated solute side. Water flows continuously until chemical potential equilibrium of the pure solvent is established. At equilibrium condition, the difference in pressure between the two sides of the dense semi-permeable membrane is equal to the osmotic pressure of the solution. To reverse the flow of water (pure solvent), a difference in external pressure substantially greater than the osmotic pressure difference is applied to the more concentrated solute of the system. The flow rate of the pure solvent molecules will drop as the pressure increases and will stop when the applied pressure numerical equals the osmotic pressure. As a result, by applying pressure will induce a opposite condition to osmosis, in which the pure solvent (water) molecules from the more concentrated part of the system will diffuse into the less concentrated part. This process phenomenon is termed as reverse osmosis (RO). The membranes have to be highly permeable to water, highly impermeable to solutes, and capable of withstanding the applied pressure without failure. RO membrane which is very dense pores are considered non-porous (approx. $0.1 - 1$ nm, MWCO over 100 Da). In RO technology, semi-permeable membrane for the desalination process applies operating pressure $(12 - 70 \text{ bar})$ on the saline water. This forces the fresh water through a semi-permeable membrane and permits the transport of water (MWCO 18 Da.) with a (very) low salinity as product water or permeate. Meanwhile, dissolved monovalent ions (salt) are rejected along with other solutes or impurities, and most organics with MWCO of greater than 150 – 250 Da. In pressure-driven membrane operations, the use of both concentrates and permeate streams leaving the same membrane unit is another key issue. In most operations, while the product stream called permeate is collected, the other variant known as concentrate or brine is discharged as waste in membrane desalination processes **[14].** The comparison of various kinds of pressure-driven membrane technologies is visualized in Figure 1 **[15].**

Figure 1. Various pressure-driven membrane processes [**15**].

Selected Applications Of Pressure-Driven Membrane Separation

A hydrostatic pressure gradient (also known as transmembrane pressure) is the driving force used to achieve the desired hydrodynamic flow across the membrane (and a deposited layer that may develop during the filtration process) and concentration gradient of the liquids **[16].** As mentioned earlier, selected applications of

membranes in this review are removing bacteria, and recovering high protein and low fat from skim milk by microfiltration (MF) membrane, separating and/or concentrating protease enzyme by ultrafiltration (UF) membrane, separating and/or concentrating valuable component in corn steeping water by nanofiltration (NF) membrane, and purifying or desalting brackish and sea waters by reverse osmosis (RO membrane. A spacermembrane-support plate-membrane-spacer sandwich (a), a photo of the Module of LabUnit M20 (b), and the schematic diagram of the membrane filtration system (c) was visualized in Figure 2 **[17].**

Figure 2. Visualization of modules used in this review as a research activity. (a). Spacer-membrane-support plate-membrane-spacer like sandwich, (b). A photo of Module of LabUnit M20 and (c). The schematic diagram of the membrane filtration system **[17].**

Removing bacteria, high protein and low fat from skim milk by microfiltration (MF)

Most of the industrial developments of membrane separation technologies in the food (and beverage), pharmaceutical and medicinal, biotechnology, chemical industries originate from the dairy industry. They have became an integral part of dairy and improvements in membrane technology due to various applications in both upstream and downstream technology **[18].** MF, which is a non-thermal method, is widely used to clarify suspensions for cell harvesting, reduce turbidity, remove the amount of bacteria, proliferate bacterial and some viruses, parasites, and spores without affecting the taste of milk in the final product, improve the microbiological safety of dairy products, and provide and extend longer shelf life without damaging sensory attributes than pasteurization. MF is also a well-established laboratory technique for producing sterile fluids without the application and treatment of heat through reducing the presence of bacteria, viruses, somatic cells, fat and lately micellar casein from skim milk **[19].** Fresh cow's milk procured in local was collected into tank. Cream in fresh cow's milk was separated by cream separator. Cream-free fresh cow's milk was further pumped through tubular ceramic MF membrane equipped in testing cell to yield two liquids that differ in their composition. The liquid that is able to pass through the tubular ceramic MF membrane is known as permeate and retained liquid on membrane surface is known as retentate or concentrate. Both, permeate contains high protein, low fat and lower microorganism count as valuable compounds compared and retentate contains lower protein, higher fat and higher microorganism compared with fresh cow's milk prior to feed to testing cell **[20].** Similar procedure is also performed on fresh cow's milk by means of flat sheet polymeric MF membrane fitted in testing modul at room temperature, flow rate 4 L/min., and transmembrane pressure (TMP) of 0.8, 1.6, 2.4, 3.2, and 4 bar **[21].** The goal of this experiment works were to evaluate the effectiveness of microfiltration (MF) membrane in removing bacterial spores from skim milk as a result of cream separation and pasteurization. Modul configuration used is flat sheet polymeric MF membrane with pore size of 0.45 μ m. Results indicated that total bacterial counts in the skim milk were determined before and after pasteurization. Results of average total bacterial counts in the skim milk determined before and after pasteurization were 5.40

x 10⁶ colony and 11.25 x 10³ colony, as tabulated in Table 1.

Table 1. Result of investigation on bacteria total count in skim milk before and after pasteurization.

Pasteurized skim milk is further microfiltered using a 0.45 µm flat sheet polymeric membrane at room temperature, flow rate 4 L/min., and TMP of 0.8, 1.6, 2.4, 3.2 and 4 bar. Flat sheet polymeric MF membrane of 0.45 μm pore size is able to remove total bacterial of 1,300, 2.300, 2.200, 1,200, and 500 colony at TMP of 0.8, 1.6, 2.4, 3.2 and 4 bar. Contents of total dissolved solids, protein and fat in permeate of flat sheet polymeric MF membrane (0.45 μm pore size) at TMP of 0.8, 1.6, 2.4, 3.2, and 4 bar is shown in Table 2. The log reduction parameter is the right method to characterize a membrane's ability to prevent bacteria passage.

Table 2. Commercial MF membrane (Fluoro polymer, 0.45 µm, Alfa Laval).

Separating and/or concentrating protease enzyme by ultrafiltration (UF)

The potential use of protease enzyme (exolite) in leather tannery industry has expanded in this past decade. The reason of success use is mainly attributed to their characteristic advantages in the efficient application, specific convenient, effective cost and friendly-environmentally. During the process, the non collagenous constituents are removed partially or completely in the various steps of processing, such as soaking, unhairing, liming, bating, etc. and extent of removal of these constituents decides the characteristics of the final leather **[22].** Microbial enzymes are replacing chemical catalysts in manufacturing chemicals, food, leather goods, pharmaceuticals, and textiles. Among proteases, alkaline proteases are employed mainly as detergent additives due to their distinctive ability to assimilate proteinaceous stains. Applications of protease for detergent industry need concentrated and cleaned enzyme to amend with detergent and to get good quality during storage and application. The enzyme is cleaner when the medium is simple, where fermented enzyme contains many sludge particles and other impurities, so enzyme has to be clarified and concentrated to get higher enzyme activity **[23].** Among the various membrane separation processes, UF membrane technology has been successfully applied to food and non food processes to improve both economic and product quality, reduce waste pollutant and save energy. Ultrafiltration (UF) is a pressure-driven membrane-based separation process, having a porous membrane enables the molecular separation of nanometer-sized species. The end goals of UF membrane application in the protease enzyme process are to replace batch to continuous culture process,

separate extra cellular enzyme from cell, purify, concentrate and separate the enzymes and other proteins in downstream process or to recover microbial products (cells and spores) present in a culture medium **[24].** Result of separation and/or concentration of protease enzyme through commercial ultrafiltration (UF) membrane (Polysulfone, 20000 MWCO, DSS) at room temperature, flow rate of ~5 L/min., and pressure of 5 bar for 150 min. was shown in Table 3.

Table 3. Effect of time on flux and enzyme activity in concentration process of protease enzyme using UF membrane (Polysulphone, 20000 MWCO, DSS).

Permeate flux value declined 30 % from before process $(87.26 \text{ L/m}^2 \cdot \text{h})$ to after process $(60.96 \text{ L/m}^2 \cdot \text{h})$. Decline of permeate flux value, that is a major limiting and obstacle factor in pressure-driven membrane processes, is caused by the build-up of rejected solute particles on the selective active membrane surface (external fouling), the deposition and adsorption of small particles at the pore entrances or within the internal pore structure of the membrane (internal fouling), concentration polarization, cake layer formation on the top membrane surface. The deposition of solute particles on or in the membranes is thought to be the major cause for the initiation of such a fouling. Compositions of protease enzyme as a result of fermentation process is usually very complex, low concentrations and heat sensitive. Cross-flow ultrafiltration (UF) membrane becomes a necessary step to separate and/or concentrate bioactive compounds prior to purification. During cross-flow UF membrane, the feed is pumped across the membrane surface. Under an applied pressure, water may penetrate through the membrane pores and the solutes may be rejected by the membrane. As the solutes are convected to the membrane surface, accumulation of the rejected solutes near the membrane surface causes the diffusion of solutes to diffuse back to the bulk stream (feed or retentate or concentrate), and results in increasing hydraulic resistance to permeate flow, as a result the permeate flux declines with time. During separation and/or concentration of protease enzyme by means of cross-flow UF membrane, it is occurred an increase of enzyme activity in concentrate of 7.3 folds from prior to process (0.035 Unit/mL.min.) to after process (0.292 Unit/mL.min.). Increase of enzyme activity is due to its presence of water mass deficit in retentate or concentrate. In consequence of water mass deficit during UF process would activate enzyme cells in retentate or concentrate, so that enzyme particle size became larger than membrane pores size. Thus, to pass enzyme particles across membrane would be obstacle **[25].**

Separating and/or concentrating valuable component in corn steeping water by nanofiltration (NF)

Fundamental separation steps through applications of membrane technology in the corn processing include are water removal (pretreatment of fresh water and wastewater treatment for recycling) and product separations (recovery of valuable and potential solids) that impact product quality and processing economics in many grains processing, such as in corn steeping water separation. Result of experiment activity showed that NF membrane is able to concentrate corn steeping water (CSW). Nevertheless, increasing the concentration time from initial to 270 min. smoothly dropped flux value from 8.65 L/m².h. to 6.66 L/m².h. (23 %) for TMP 10 bar, sharply declined from 21.63 L/m².h. to 6.44 L/m².h. (70 %) for TMP 20 bar, and sharply decreased from 22.99 L/m².h to 7.50 L/m².h. (67 %) for pressure 25 bar, respectively. This declining flux value is caused by fouling and concentration polarization on the top membrane surface. Meanwhile, increasing the concentration time from initial to 270 min. gradually increased dissolved protein content from 3.312 to 4.036 mg/g (21.8 %)

for TMP 10 bar, from 3.428 to 3.973 mg/g (15. 9 %) for TMP 20 bar, and from 3.055 to 4.116 mg/g (25.8 %) for TMP 25 bar, respectively. Effect of concentration time on flux value and dissolved protein in concentrating corn steeping water (CSW) through nanofiltration (NF) membrane at room temperature, pump motor frequency 10 Hz, and TMP of 10, 20 and 25 bar for 270 min.) is tabulated in Table 4 [**26]**.

Table 4. Effect of concentration time on flux value and dissolved protein in concentrating corn steeping water (CSW) through nanofiltration (NF) membrane (Thin Film Composite on Polyester, DSS) at room temperature, pump motor frequency 10 Hz, and TMP of 10, 20 and 25 bar for 270 min. **[26].**

Purifying or desalting brackish and sea water by reverse osmosis (RO)

RO is one of the families of pressure-driven membrane separation (PDMS) processes that is potentially applicable to desalt and purify sea water and brackish water to produce portable, fresh and clean water, to clean effluents, and recover valuable components. Desalination by means of PDMS process plays an important role in water sustainability for many countries around the world, including Indonesia as the world's largest island or archipelago country **[27].** In RO, when water molecules reach the membrane surface, they actually dissolve into the membrane material and then diffuse via it at a rate proportional to the net pressure differential across the membrane. Dissolved solutes diffuse through a RO membrane at slower rates and are less soluble in membrane materials due to their physical and chemical properties as ions in fluid. Result of separation and/or purification treated brackish water by MF using commercial composite RO membranes (HR-98-PP, DSS, Denmark) at room temperature, flow rate of \sim 5 L/min., and TMP of 20 bar gave permeate flux of 20.78 $L/m²$.h. The transport of component across a membrane is commonly expressed as component rejection. While, observer rejection (R_{obs}) is determined experimentally for each component or species in the feed, by sampling the feed and permeate at the same time and analyzing that component **[28].** Mechanism of Reverse Osmosis was visualized in Figure 3 **[29].**

Figure 3. Mechanism of Reverse Osmosis **[29].**

Pretreatment step of fresh sea water and brackish water include flocculation-coagulation, sedimentation, multi media filtration, single medium filtration, activated carbon treatment and cartridge filtration. Pretreatment of

fresh sea water and brackish water has essential role to reduce the resistance of the gel layer (increase porosity, reduce thickness), prevent or minimize membrane fouling or damage by harmful brackish water and sea water components or oxidizing agents. Composition of dissolved ions in fresh brackish water, treated brackish water by MF and permeate, and rejection as a result of separation and/or purification by commercial composite (DSS) RO membranes at flow rate of ~5 L/min., room temperature and TMP of 20 bar, and membrane performance were represented in Tables 5a and 5b, respectively. Table 5a and 5b showed that dissolved ions concentration in fresh brackish water after through pretreatment process had drop. This matter is caused by successfully operation of brackish water pretreatment. In coagulation-floculation process, addition to FeCl₃ coagulant and polyacrylic amide into brackish water would bind physico-chemically inter fine particles solids, so that it is formed tough and heavy flocs. As a consequency of gravity force, settling of flocs is occurred to form fine deposit. Fine deposit was then separated, while treated brackish water was subsequent flown to multi layer filter system, activated carbon filter, and cartridge filter to get treated brackish water. Treated brackish water by MF was then purified by means of HF or RO membrane in module scale to get target permeate **[30].**

Composition of dissolved ions in fresh sea water, treated sea water by MF and permeate, and rejection as a result of separation and/or purification by commercial composite RO membranes at room temperature, flow rate of ~5 L/min., and TMP of 40 bar, and membrane performance were represented in Table 5b. Result of separation and/or purification treated sea water by using RO commercial composite membrane (DSS) at room temperature, flow rate of \sim 5 L/min., and TMP of 40 bar gave permeate flux of 28.07 L/m².h. On the other hand, dissolved ions, such as Na⁺, K⁺, Ca²⁺, Mg²⁺, Cl⁻, HCO₃²⁻, SO₄²⁻ that passes across the commercial composite membrane (DSS) into the permeate was subsequent 6.4% , 5.8% , 4.9% , 1.6% , 1.2% , 9.6% , and 1.6 %. Its presence of dissolved ions that penetrates through both membranes due to its occurrence interaction inter-particles and interaction between particles and membrane itself **[30].**

Table 5b. Performance result of commercial RO membrane (Thin film composite on Polypropylene, DSS) using sea water at room temperature, flow rate ~5 L/min., and TMP 40 bar for 210 min. **[30].**

Benefits and Drawbacks

The main benefits of pressure-driven membrane separation technology when compared to other conventional unit operations in (bio)chemical engineering are related to this unique separation principle (the transport selectivity of the membrane). A few potential advantages and possibility of avoiding toxic solvents in the use of pressure-driven membranes separation technology (MF, UF, NF or RO) is related to athermal process, the absence of change in phase (the liquids remain liquids) and no chemical agents of the fluid during the membrane separation process when compared to other conventional separation techniques (evaporation or freeze-drying). Separation can be conducted under mild operation conditions (no heat deterioration of the products) which makes it a suitable technique in food and biotechnology industry as a routine processing tool, smooth operation, less space requirement, and greener environmental friendliness. To achieve the wanted effect, it is sometimes necessary to combine, integrate and hybrid membranes technique with other separation processes technique to adjust and increase capacity (just add more modules) and membrane properties, and enhance membrane performance with their synergistic effects **[31].**

Although, there is a number of excellent benefits, these pressure-driven functional polymeric membrane separation technology have some principal problem arising, such as fouling and concentration polarization. Fouling is a term generally used to describe the undesirable accumulation of various foulant onto active membrane or within its pores. The presence of a deposit layer introduces additional resistance to permeate flow while the pore clogging changes the effective distribution of membrane pore size. Various fouling patterns in pore blocking mechanisms laws developed for empirical dead-end filtration are a function of the particles size and shape in relation to the membrane pore size distribution covering complete pore blocking law, intermediate (partial) pore blocking law (long-term adsorption), cake formation law (boundary layer resistance), and standard (internal) pore blocking or constriction (solute direct adsorption). When a mixture in fluid is brought to a membrane surface by any driving force, it can occur an accumulation of the less permeable species and a depletion of the more permeable components in the boundary layer as a result of dropping in the available driving force, referred to as concentration polarization (CP). Concentration polarization (CP) raises the osmotic pressure at the membrane surface and concentration of soluble specious in the boundary layer exceeds their solubility limits, which causes a dropping in water flux. As a consequence of CP, it reduces the overall efficiency of separation and raises the costs of capital and operation **[32].**

CONCLUSIONS AND PROSPECTIVE VIEW

Although, functional polymeric membrane can be viewed as a narrow sub division, however it is able to be applied in broader field, such as desalination brackish and sea water, biotechnology (pharmaceutical, enzyme, fermentation), food and beverage, dairy, sugars, pulp and paper, water treatment.

As mentioned in benefits and drawbacks earlier, pressure-driven membrane separation technology has met the requirements of green process engineering toward the realization of substantial improvements in chemical, manufacturing and processing aspects, and toward achieving sustainable industrial development assisting in the pursuit of the aims of total raw materials utilization, low energy consumption and zero liquid discharge (ZLD).

To achieve various demands for higher degrees of separation, purification, fractionation, clarification and concentration of desirable and target components in fluid, these membrane-based separation unit operations methods can be operated separately or integrity and conjunction with other conventional separation unit operation, in which substantial reduction in overall energy demand, environmental footprint, and process

hazards has already been accomplished.

The new field of functional polymeric membranes application is continually offering challenges in basic scientific research and very basic knowledge in membrane separation technology, and performing a wider range of application in industry, particulary marine-oriented sources, agro-industry sources to anticipate the global challenge through increasing added value of marine processed products and agro-industry products for the economic growth of the country.

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