Membranes separation processes

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Outline

1. Introduction
2. Overview of membrane processes
3. Modules and process design
4. Main phenomena
5. Membrane transport theory and modeling

References

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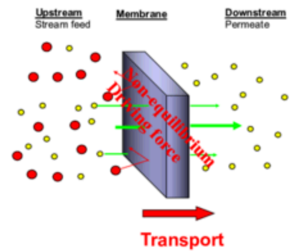
1. Introduction

Classification of separation processes

Type	Feed	Principle	Unit operation
Mechanical-physical	Heterogeneous phase	Separation of solids, liquids or gases by mechanical means	Suspensions (filtration settling, cyclones, ...) Mixed Solids (sieving, criblages, magnetic sep...)
Mass transfer	Homogeneous phase	Mass transfer between 2 phases in direct contact	Energetic agent (distillation, evaporation, cristallisation, ...) Material agent (extraction, absorption, stripping, ...)
Mass transfer	Homogeneous phase	Mass transfer between 2 phases separated by a barrier	**Membranes processes** Others (chromatography, spectrométrie de masses, ...

Separation with a membrane



Benefits and Drawbacks

- ☺ Can be carried out under midle conditions (ambiant temperature) ⇒ thermosensitive compounds, ...
- ☺ No additives are required
- ☺ No phase change⇒ low energy consomption
- ☺ Can be carried out continuously
- ☺ Easy up-scaling

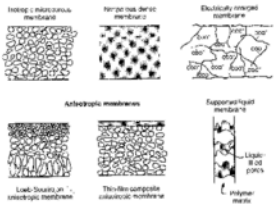
- ☹ Fouling
- ☹ Partial selectivity
- ☹ Limited lifespan(mechanical stability is reduced, damaging due to the periodic chemical washing)
- ☹ Relative high flow rates are required ⇒ pumping cost

1.1 The membranes

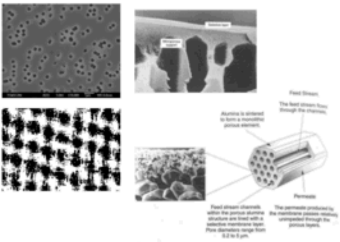
Definition of a membrane

- A Permselective Membrane: a selective barrier between 2 phases allowing specific mass transfers.
- The membrane has the ability to transport one component more readily than other
- The selectiv transfer through the membrane may be due to differences in physical and/or chemical properties between the membrane and the permeating components (species size, affinity...)

Types of membranes



Types of membranes



Membranes materials

- Organic membranes (> 80%)
  - Polymers or macromolecules: from cellulose, polyamides, polyimide, polysulfone, polyolfins and fluoro polymers, derived acrylic compounds, others (polycarbonates,…)
  - ⊕ Easy Carrying out
  - ⊕ Available in a large pore size range
  - ⊕ Low cost
  - ⊖ Chemical, mechanical and thermal stability
- Inorganic membranes
  - Ceramic, carbon, metal
  - ⊕ Chemical, mechanical and thermal stability
  - ⊖ Only available in plan and tubular geometries
  - ⊖ Small pore size membrane not available
  - ⊖ breakable
  - ⊖ Cost

2. Overview of membrane processes

Vocabulary used

- **Feed, Residue** or **retentate** and **permeate**
- **Pressure drop**

When a liquid flows through a pipe a pressure drop i observed across the flow geometry due to friction with the wall

- **Transmembrane pressure**
- **Permeate flux**, J ($m^3.s^{-1}.m^{-2}$ or $m.s^{-1}$)

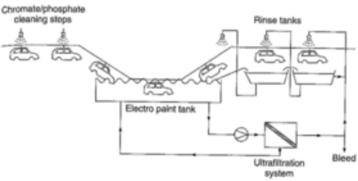
Permeate flow rate par membrane area

Mean permeate velocity

- **Retention or rejection coefficient** $TR = \frac{c_0 - c_p}{c_0} = 1 - \frac{c_p}{c_0}$
- **Conversion rate** $Y = \frac{Q_p}{Q_0}$
- **Selectivity factor** $a_{AB} = \frac{c_{A,permeate}/c_{B,permeate}}{c_{A,feed}/c_{B,feed}}$

2.1 Pressure driven membrane processes

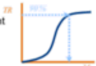
Applications of ultrafiltration: electrocoat paint



Ultrafiltration (UF)

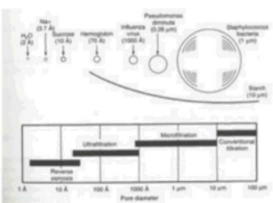
Cross section of an ultrafiltration polysulfone membrane

- Suitable for retaining macromolecules and colloids
- Polymeric and ceramic membranes materials
- Asymetric macro or meso-porous membranes (1 nm< *pore sizes* <100 nm)
- Separation based on size and shape of the solutes relative to the pore size (sieving mechanism)
- Membrane selectivity is characterized with the « Cut-off »: solute molecular weight the retention of which is 90%
- Applications:
  - Dairy (milk, whey, cheese making)
  - Food (potato starch and protein)
  - Metallurgy (oil-water emulsion, electropaint recovery)
  - Pharmaceutical (enzyme, antibiotocs, pyrogens)
  - Water treatment



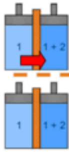
Microfiltration (MF)

- Suitable for retaining suspensions and emulsions
- Separation of particles
- Symmetric and asymetric porous membranes (100 nm < *pore sizes* < 10 µm)
- Polymeric and ceramic membranes materials
- Applied pressure low (< 2 bar)
- Separation based on particle size (sieving mechanism)
- Applications:
  - Cold sterilisation of beverages and pharmaceuticals
  - Cell harvesting
  - Clarification of fruit juice, wine and beer
  - Ultrapure water in the semiconductor industry
  - Waste-water treatment



Reverse osmosis, ultrafiltration, microfiltration and conventional filtration are related processes differing principally in the average pore diameter of the membrane (porous, dense where discrete pores do not exist)

Osmotic pressure



$$\mu_{1,gauche} = \mu_1^* > \mu_{1,droite} = \mu_1^* + RT\ln(\gamma_1\tilde{x}_1) + \tilde{V}_1(p - p_{ref})$$

Osmosis

$$\mu_{1,gauche} = \mu_{1,droite}$$

$$\mu_1^* = \mu_1^* + RT\ln(\gamma_1\tilde{x}_1) + \tilde{V}_1\Pi \Rightarrow \Pi = -\frac{RT\ln(\gamma_1\tilde{x}_1)}{\tilde{V}_1}$$

$$\ln(x_1) = \ln(1 - x_2) = 0 - x_2 - \frac{x_2^2}{2} - \ldots \approx -x_2 \Rightarrow \Pi = +\frac{RT\tilde{x}_2}{\tilde{V}_1}$$

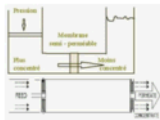
$$\tilde{x}_2 = \frac{n_2}{n_1 + n_2} \approx \frac{n_2}{n_1} \Rightarrow \Pi = \frac{RTn_2}{\tilde{V}_1 n_1} = \frac{RTn_2}{V} = RTc_2$$

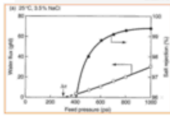
$\Pi = iRTc$ **The Van't Hoff's law**

(ideal case, dilute solutions)

Reverse osmosis

- Suitable for retaining salts
- Asymetric or compodite dense membrane (*pore size* <2 nm)
- Polymeric membranes: cellulose triacetate, aromatic polyamide, polyamide and poly (ether urea)
- Applied pressure high (30-80 bar)
- The solvant (mainly water) goes through the membrane whilst the salts are retained
- The separation principle is based on solution-diffusion
- Applications:
  - Seawater desalination (salt concentration of 3.2-4.0 %)
  - Brackish water desalination (2000-10000 mg/L)
  - production of ultrapure water
  - Waste water treatment (recovery of nickel from nickel-plating baths…
  - Organic solvent separation (séparation de mélanges organiques)





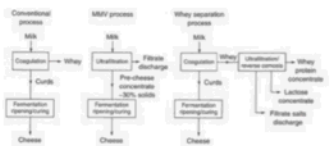
Desalination Membrane SW-30 (FilmTec - Dow) Cadotte et coll. (1980)

Nanofiltration (NF)

- Microporous composite membrane (*pore size* <2 nm)
- Membrane material: polyamide
- Applied pressure relatively high (10-25 bar)
- A complex separation principle:
  - Solution-diffusion
  - Sieven mechanism
  - Exclusion of Donnan

  Neutral Membranes : Na_2SO_4>$CaCl_2$>NaCl
  Anionic Membranes : $CaCl_2$>NaCl>Na_2SO_4
  Cationic Membranes : Na_2SO_4>NaCl>$CaCl_2$
- High retention of bivalent ions and some non-ionic organic compounds (*M*>300 g/mol)
- Low retention of monovalent ions and some non-ionic organic compounds (*M*<300 g/mol)
- Applications:
  - Desalination of brackish water
  - Removal of micropollutents
  - Water softening
  - Waste water treatment
  - Retention of dyes (textiles industry)

Applications of ultrafiltration: cheese making



Simplified flow shamatic showing the traditional cheese production method and two new methods using ultrafiltration to increase the recycle of useful product

2.2 Concentration driven membrane processes

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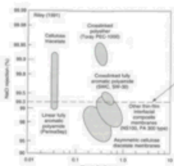
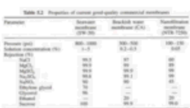
Applications of reverse osmosis: seawater desalination

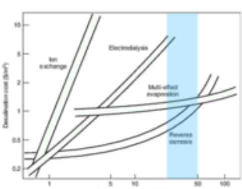
Desalinizadora de El Prat (Barcelone):
Agbar - Degremont
200 000 m³/day
4,5 millions of habitants
230 millions of euros

Applications of reverse osmosis: seawater desalination

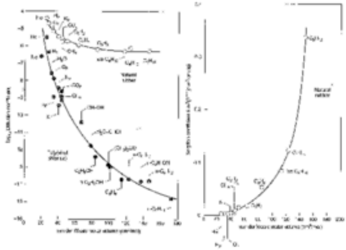




Applications of reverse osmosis: seawater desalination



Gas separation



Gas separation

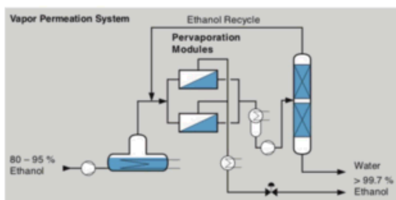
- Fractionation of a gas mixture based on partial transport under pressure
- Transport mechanisms:
  - Knudsen diffusion (porous membrane, δ_p<100 nm): mean free path of the gas molecules greater than the pore diameter
  - Molecular Sieve ($\delta_p \approx$ 5-20 Å)
  - Solution + diffusion (non-porous membranes): a balance must be found between permeability and selectivity

$$J_i = \frac{\mathcal{D}_i K_i^G (\Delta p_i)}{l} = \frac{\mathcal{P}_i (\Delta p_i)}{l}$$

$$\alpha_{ij} = \frac{\mathcal{P}_i}{\mathcal{P}_j} = \frac{\mathcal{D}_i K_i^G}{\mathcal{D}_j K_j^G}$$

- Applications
  - Enrichment of uranium hexafluoride (separation factor=1,0064)
  - Air separation to obtained oxygen-enriched air and nitrogen-enriched air
  - Dehydration of natural gas, air conditioning and drying of compressed air
  - SO_2, CO_2 and NOx removal from smoke and flue gas

Pervaporation Dehydration of (bio)ethanol



Pervaporation

- A liquid mixture is in contact with the membrane on the feed at atmospheric pressure and a low vapour pressure existing on the permeate side thanks to a vacuum pump
- Non-porous Composite membranes with an elastomer or glassy polymeric top layer
- Process involving a sequence of 3 steps:

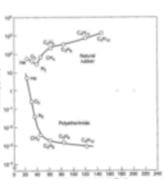
Selective sorption into the membrane on the feed side

Selective diffusion through the membrane

Desorption into a vapour phase on the permeate side

- Applications:
  - Dehydration of organic solvent
  - Removal of volatil organic compounds from water (alcohol, aromatics, chlorinated hydrocarbons
  - Separation of isomers (o-xylène, m-xylène, p-xylène…)

Gas separation



Process	Application	Comments
Established processes		
Oxygen/nitrogen	Nitrogen from air	Processes are all well developed. Only incremental improvements in performance expected
Hydrogen/methane; hydrogen/nitrogen; hydrogen/carbon monoxide	Hydrogen recovery; ammonia plants and refineries	
Water/air	Drying compressed air	
Developing processes		
VOC/air	Air pollution control applications	Several applications being developed. Significant growth expected as the process becomes accepted
Light hydrocarbons from nitrogen or hydrogen	Reactor purge gas, petrochemical process streams, refinery waste gas	Application is expanding rapidly
Carbon dioxide/methane	Carbon dioxide from natural gas	Many plants installed but better membranes are required to change market economics significantly
To-be-developed processes		
C_{3+} hydrocarbons/methane	NGL recovery from natural gas	Field trials and demonstration systems under way. Potential market is large
Hydrogen sulfide, water/methane	Natural gas treatment	Niche applications, difficult for membranes to compete with existing technology
Oxygen/nitrogen	Oxygen enriched air	Requires better membranes to become commercial. Size of ultimate market will depend on properties of membranes developed. Could be very large
Organic vapor mixtures	Separation of organic mixtures in refineries and petrochemical plants	Requires better membranes and modules. Potential size of application is large

Dialysis

- Suitable for separating low molecular weight components from those of high molecular weight
- Homogeneous membranes (10 μm < *thickness* < 100 μm)
- Hydrophilic (regenerated cellulose) and hydrophobic (cellulose acetate, poly vinyl alcohol…) materials
- Separation based on difference in diffusion rate, solution-diffusion

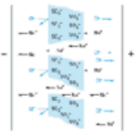
An optimum must be found between the diffusion rate and swelling

- Applications:
  - Hemodialysis (removal of toxic substances from blood)
  - Alcohol reduction in beer
  - Desalination of enzymes and coenzymes
  - Alkali recovery in pulp and paper industry

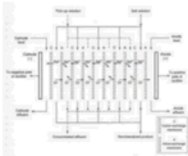
2.3 Electrically driven membrane processes

Electrodialysis

- Use of an electrically charged membranes to remove ions from aqueous solution when a direct current is applied



Cation-exchange Membrane



- Several hundreds of cell pairs are assembled in a stack. In this way, the applied driving force is used very effectively

Electrodialysis

- Cation-exchange and anion-exchange membranes
- Nonporous membrane (100 μm < thickness < 500 μm)
- Membrane material: crosslinked copolymers based on divinylbenzene with polystyrene or polyvinylpyridine
- Applications

- Desalination of water

- Desalination in food and pharmaceutical industry (acids removal in fruits juice, desalination of sweet juice…)

- Separation of amino-acids

- Production of salt

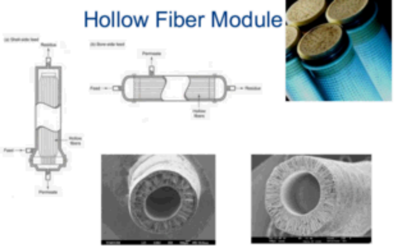
State of the art

Category	Process	Sates
Developed industrial membrane separation technologies	Microfiltration Ultrafiltration Reverse osmosis Electrodialysis	Well-established unit operation. No major breakthroughs seem imminent
Developing industrial membrane separation technologies	Gas separation Pervaporation	A number of plants have been installed. Market size and number of applications served and expanding
Te be developed industrial membrane separation technologies	Carrier facilitated transport Membrane contactors	Major problem remains to be solved before industrial system will be installed on a large scale

3. Modules and process design

3.1 Modules design

Hollow Fiber Module

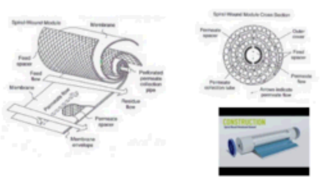


- Two types of modules: feed solution can enter inside the fiber (« inside-out ») or on the outside (« outside-in »)
- The packing density (fiber-diameter-dependent) : 500 – 30 000 m^2.m^{-3}

Tubular Module

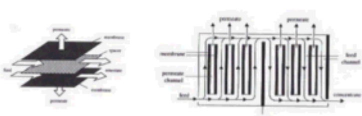
- Not self-supporting membranes: they are placed inside a porous stainless steel, ceramic or plastic tube with the diameter of the tube being more than 10 mm
- The packing density : 10 – 400 m^2.m^{-3}

Spiral-wound Module



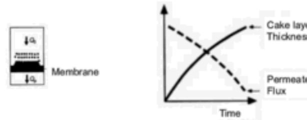
- The Next logical step from a flat membrane.
- A plate-frame system wrapped around a central collection pipe in a similar fashion to a sandwich roll
- The packing density: 300 – 1000 m^2.m^{-3}

Plate-and-frame Module



- Configuration closest to the flat membranes used in laboratory Sets of 2 membranes are placed in a sandwich-life fashion with their feed sides facing each other
- The packing density : 100 – 400 m^2.m^{-3}

Dead-end operation



- The feed is forced through the membrane
- The concentration of rejected components in the feed increases
- Flux decline because of cake grow
- Used very frequently in microfiltration

3.2 System design

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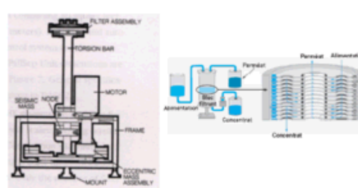
Application	Module type
Reverse osmosis-seawater	Spiral-wound modules. Only one hollow fibers producer remains
Reverse osmosis industrial and brackish water	Spiral-wound modules used almost exclusively; fine fiber too susceptible to scaling and fouling
Ultrafiltration	Tubular, capillary (limited to highly fouling feeds) and spiral-wound modules (clean feeds) all used.
Gas separation	Hollow fibers for high volume applications with low flux, low selectivity membranes in which concentration polarization is easily controlled Spiral wound when fluxes are higher, feed gases more contaminated and concentration polarization a problem (natural gas separation, vapor permeation)
Pervaporation	Most pervaporation systems are small, plate-and-frame systems are used Spiral-wound and capillary modules being introduced

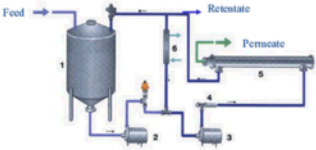
	Tubular	Plate-and-frame	Spiral-wound	Hollow fiber
Packing density	Low	→	→	Very high
Investment US $/m^2	High 50-200	50-200	5-10	Low 5-20
Fouling tendency	Low	→	→	Very high
Cleaning	Good	→	→	Poor
Membrane replacement	Yes/no	Yes	No	No

Vibrating Modules



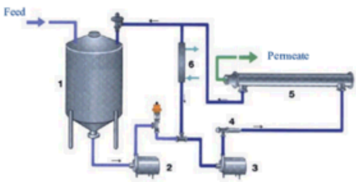
- Vibration of the membrane creates interior agitation directly at the membrane surface.
- Suitable for extremely concentrated visous solutions

Continuous system



- A single-pass system: the feed solution passes only once through the module
- A recirculation system: the feed is pressured by a pump and allowed to pass several times through the module. Suitable in case of severe fouling and concentration polarisation

Batch System

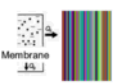


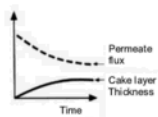
Batch with a feed

Batch System

- A batch system can be used for small-scale applications
- Volume of the feed decreases with time
- Recirculation system: loop

Cross-flow operation





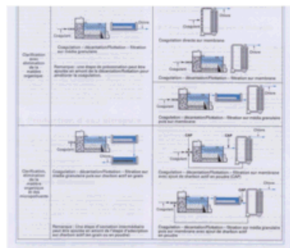
- The feed flows parallel to the membrane surface
- Reduced concentration polarization
- Lower fouling tendency
- A cross-flow operation is preferred for industrial applications

Some examples

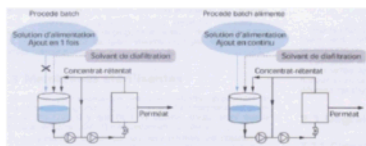
- Feed solution enters inside the fiber
- High feed pressure
- Use of a booster pump

- Feed solution enters on the outside
- Low permeate pressure
- Use of a succion pump in the permeate side

Hybrid system

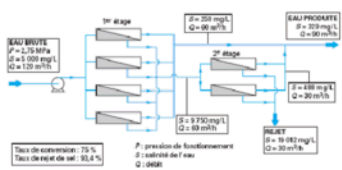


Diafiltration system



- Diafiltration can be considered as a continuous stirred tank reactor with a membrane placed in the outlet stream
- The volume in the feed remains constant because solvent is added at a rate equal to the permeation rate

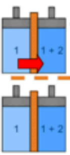
Mono or multi-stage system



4. Main phenomena. Dimensioning elements

4.1 Main phenomena

Osmotic pressure



$$\mu_{1,gauche} = \mu_1^* > \mu_{1,droite} = \mu_1^* + RT\ln(\gamma_1 \tilde{x}_1) + \tilde{V}_1(p - p_{ref})$$

Osmosis

$$\mu_{1,gauche} = \mu_{1,droite}$$

$$\mu_1^* = \mu_1^* + RT\ln(\gamma_1 \tilde{x}_1) + \tilde{V}_1 \Pi \Rightarrow \Pi = -\frac{RT\ln(\gamma_1 \tilde{x}_1)}{\tilde{V}_1}$$

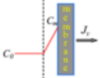
$$\ln(x_1) = \ln(1 - x_2) = 0 - x_2 - \frac{x_2^2}{2} - \ldots \approx -x_2 \Rightarrow \Pi = +\frac{RT\tilde{x}_2}{\tilde{V}_1}$$

$$\tilde{x}_2 = \frac{n_2}{n_1 + n_2} \approx \frac{n_2}{n_1} \Rightarrow \Pi = \frac{RTn_2}{\tilde{V}_1 n_1} = \frac{RTn_2}{V} = RTc_2$$

$\Pi = iRTc$ **The Van't Hoff's law**

(ideal case, dilute solutions)

Concentration Polarisation



Factor of polarisation $\gamma = \frac{c_m}{c_0}$

- When a driving force acts on the feed solution, the solute is partly retained by the membrane whereas the solvent permeates.
- Retained solute accumulates at the membrane surface where their concentration will gradually increase. The layer of solution immediately adjacent to the membrane surface becomes depletedin the permeating solute end enriched in the solute
- Consequences
  - Flux will be lower
  - Retention can be lower: low molecular weignt solutes such as salts
  - Retention can be higher : in the case of mixtures of macromolecule augmentation de la pression osmotique $TR_{obs} = 1 - \frac{c_p}{c_0}$ $TR_{int} = 1 - \frac{c_p}{c_m}$
  - Osmotic pressure will be higher

Concentration Polarisation

Membrane operation	Influence of concentration polarisation
Reverse osmosis	moderate
Ultrafiltration	strong
Microfiltration	strong
Gas separation	Very low
pervaporation	low
Electrodialysis	strong
Dialysis	low

- Influence of flow conditions: velocity, viscosity, density, solute diffusion coefficient
- Use of turbulence promoters:
  - An increased mass tranfer coefficient
  - Spacer materials to separate both membranes in the feed
  - Added specific promoters

Fouling

- A very complex phenomenon
- Difficult to describe theorically
- The (ir)reversible deposition of retained particles, colloids, macromolecules…⇒ obstruction des pores
  - Adsorption
  - Pore blocking
  - precipitation
  - Cake formation
- Flux decline
- Selectivity change
- Fouling concerns mainly porous membranes, pressure driven systems
- Methods to reduce fouling: pretreatment of the feed solution, membrane properties, module and process conditions, cleaning

4.2 Influence of operating conditions. Main trends

Permeate flux

In the case of pure solvant Influence of hydrodynamic resistance (a membrane constant) R_M

In the case of a mixture

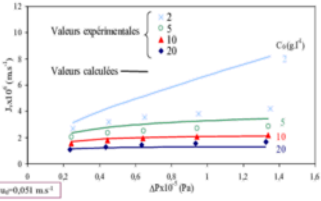
Zone 1 : linear variation
Influence of ΔP = influence of hydrodynamic resistance R_M

Zone 2 : limiting flux
No influence of ΔP = influence of mass layer resistance R_p

Influence of concentration

Hollow fiber Module– Dextran T500 [Yeh 1996]

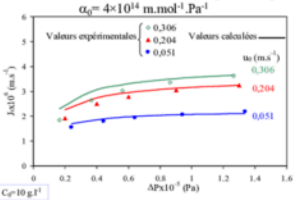
$\alpha_0 = 4\times10^{14}$ m.mol^{-1}.Pa^{-1}



Influence of axial velocity u_0

Hollow fiber Module– Dextran T500 [Yeh 1996]

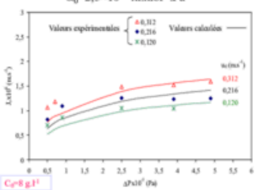
$\alpha_0 = 4\times10^{14}$ m.mol^{-1}.Pa^{-1}



Influence of axial velocity u_0

Tubular Membrane (1,2 m) – Dextran T500

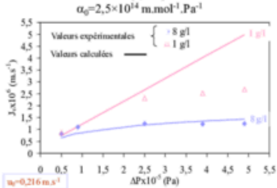
$\alpha_0 = 2{,}5\times10^{14}$ m.mol^{-1}.Pa^{-1}



Influence of pressure

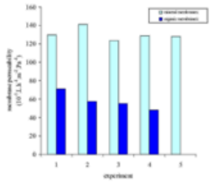
Tubular Membrane (1,2 m) – Dextran T500

$\alpha_0 = 2{,}5\times10^{14}$ m.mol^{-1}.Pa^{-1}



Membrane cleaning

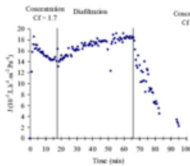
- Chemical cleaning
- Backflush cleaning



The permeability

$$J_v = L_p \, \Delta P$$

Influence of process

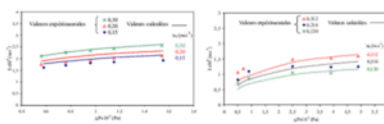


Lipogelosome mixture. Vibrating membranes.

Influence of membrane length

Tubular Membrane – Dextran T500

C_0=8 g.l^{-1}



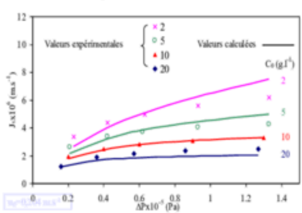
L=0,4 m

L=1,2 m

Influence of concentration

Hollow fiber module– Polyvinylpyrrolidone PVP-360 [Yeh 1993]

$\alpha_0 = 2{,}5\times10^{14}$ m.mol^{-1}.Pa^{-1}



Membrane cleaning

The permeate flux

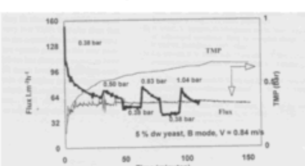
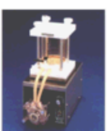


Fig. 7. Constant flux operation with slightly rising TMP compared with controlled TMP as marked.

4.3 Dimensioning elements



200 ml - 0.001 m²

2 m³/h - 50 m²

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Preliminary step

- Definition of aims
- Analysis of the feed to be treated
  - sizes of components
  - Concentration of components
  - Chemical behavior
  - Physical and chemical characteristics
- First experiments on a labscale
  - choice of the membrane
  - (material, cutt-off)

Experiments on a pilot scale

- The permeate flux as a function of:
  - transmembrane pressure
  - axial velocity
  - time
  - temperature
- Determination of the suitable operating conditions
- Design of the process
  - Batch system
  - Continuous system
  - Mono or multi-stage system

Membrane area

- Calculation of membrane area A

$$A = \frac{V}{t\,J}$$ (Batch)

$$A = \frac{Q_E\,C_E - Q_S\,C_S}{J\,C_P}$$ (Continuous)

- Scaling-up
  - the liquid path is kept constant
  - membrane area is proportional to the feed volume or to the feed flow rate
  - the axial velocity is kept constant
  - the transmembrane pressure is kept constant
  - the treatment duration is kept constant

5. Membrane transport theory and modeling

Centrale Méditerranée

5.1 Membrane transport theory

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The solution-diffusion model

$$J_i = -L_i \frac{d\mu_i}{dx}$$

$$d\mu_i = RT\,d\ln(\gamma_i \tilde{x}_i) + \tilde{V}_i\,dp$$ (influence of composition and pressure)

$$\mu_i = \mu_i^{\circ} + RT\ln(\gamma_i \tilde{x}_i) + \tilde{V}_i (p - p_{i,ref})$$ (*incompressible media*: *liquid phase, membrane*

$$\mu_i = \mu_i^{\circ} + RT\ln(\gamma_i \tilde{x}_i) + RT\ln\frac{p_i}{p_{i,ref}}$$ (*ideal* **gas**)

<u>Assumptions:</u>

- the fluids on either side of the membrane are in equilibrium with the membrane material at the interface
- the pressure throughout the membrane is constant at the highest value

$$J_i = -L_i \frac{d\mu_i}{dx} = -\frac{L_i RT}{\tilde{x}_i}\frac{d\tilde{x}_i}{dx}$$

$$c_i = M_i \tilde{\rho} \cdot \tilde{x}_i \Rightarrow J_i = -\frac{L_i RT}{c_i}\frac{dc_i}{dx} = -\mathcal{D}_i \frac{dc_i}{dx} \Rightarrow J_i = \frac{\mathcal{D}_i \left(c_{i,0,(m)} - c_{i,p,(m)}\right)}{l}$$