Standardization for Assessment of Hollow Fiber Membrane and Module for Degassing

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Membranes for Vapor Permeation

Utilization of hydrophobicity of the microporous membranes
Vapor permeation without liquid leak

■ Laplace-Young equation : $$P = 2\sigma \cos\theta / r$$

p = breakthrough pressure
θ = contact angle
σ = surface tension of the water
r = radius of pore in microporous membrane

■ **breakthrough pressure for PP membrane with pore size of 0.1 ㎛ in contact with water : 150 psig**

Methods for removing dissolved oxygen

	Heating Method
Physical method	**Vacuum Method**
	Membrane Method
Chemical method	**Hydrazine**
	Sodium sulfite

Advantages of Membrane Method

- High contact area
- High Efficiency
- Less pollution problem

Application for Vapor Permeation

- Degassing from water
  - Rinse water after photoresist etching in semiconductor industry
  - Cooling water for nuclear power plant
  - Water for alcoholic beverage
  - Ozonized water
- Blood oxygenator
- Membrane distillation
- Pervaporation
- VOC removal from water
- Breathing wear : Gore Tex
- Packaging film for food

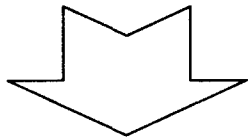
Principles

■ Henry's Law

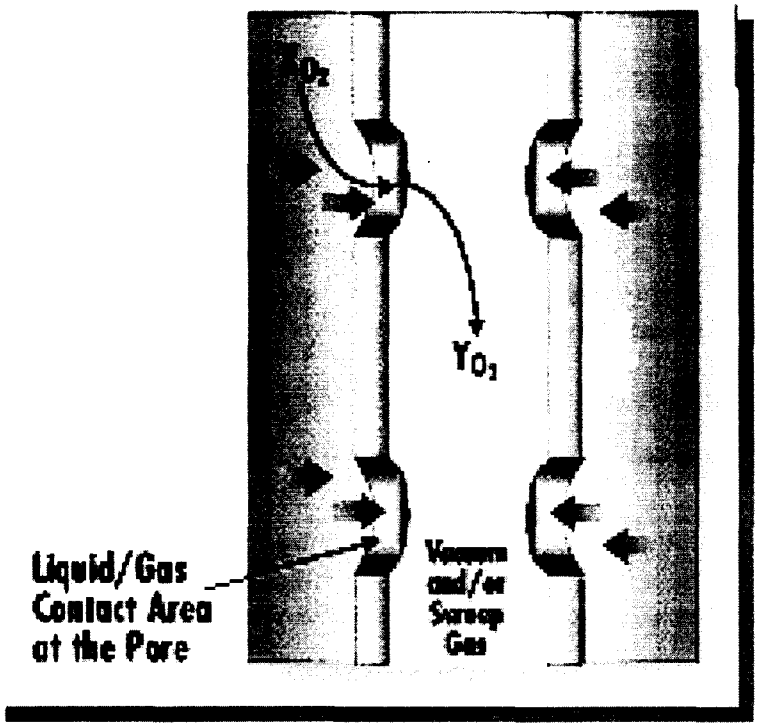
$$p = Hx$$

p = gas partial pressure
H = Henry's law coefficient
x = concentration of dissolved solute at equilibrium

■ By changing the partial pressure of the gas



Remove from or dissolve gas into water



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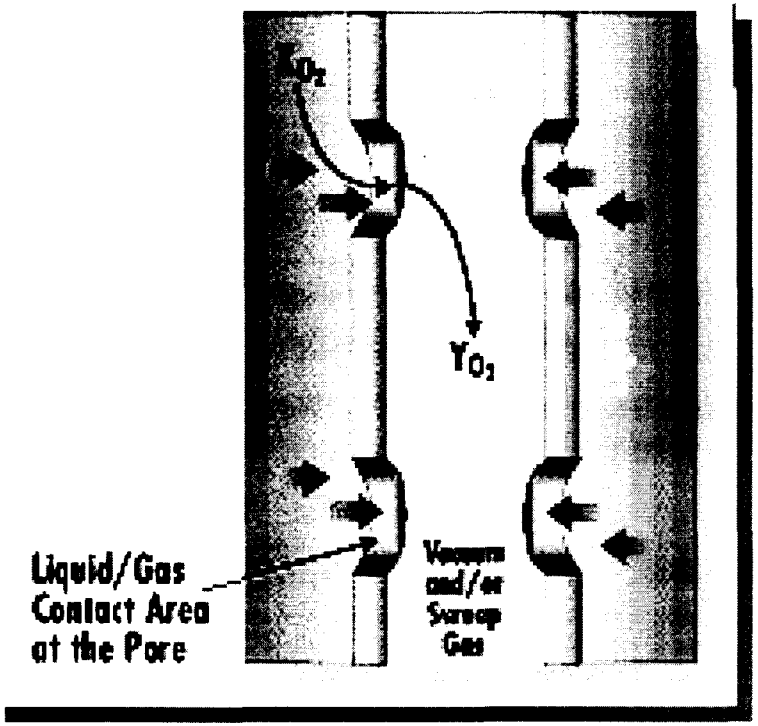


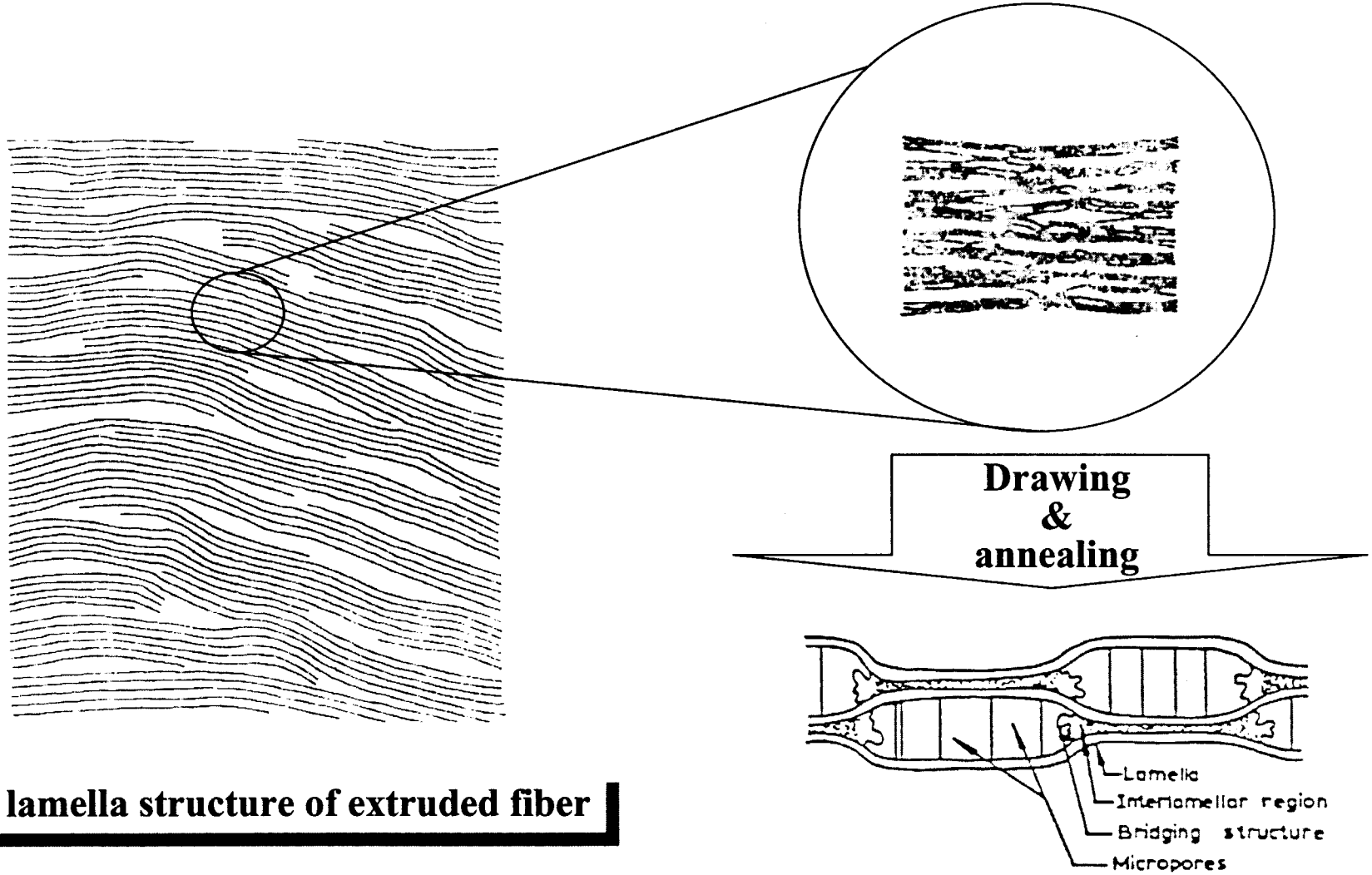
■ By changing the partial pressure of the gas

Remove from or dissolve gas into water

Methods for preaparation of membrane for degassing from semicrystalline polymer

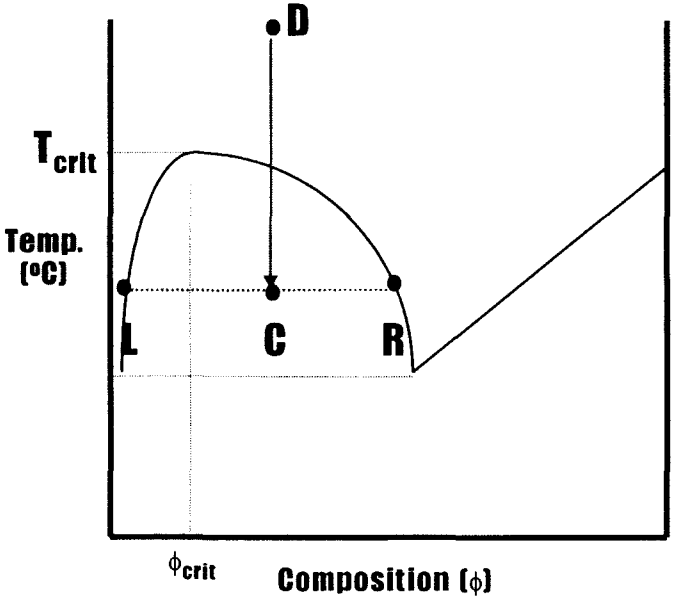
- Melt Spinning and Cold Stretching (MSCS) Method
  - Spinning or extrusion of oriented dense membrane
  - Annealing to enhance the crystalline structure
  - Stretching at room temperature : micropore formation
  - Annealing to relax the stress
- Thermally-Induced Phase Separation (TIPS) Method
  - Melt blending of polymer and diluent
  - Spinning or extrusion
  - Controlled cooling during take-up to induce phase separati
  - Removal of diluent : micropore formation
  - Drying

Mechanism of MSCS method



Piled lamella structure of extruded fiber

Mechanism of TIPS method



D

Homogeneous melt solution

cooling into liquid-liquid phase separation region

C

Phase separation

Holding at Tc

Droplets growth

Polymer-rich phase (ϕ_R)

R

Polymer-lean phase (ϕ_L)

L

Comparison of each method

	MSCS method	*TIPS method*
Material & Characteristics	• pure polymer • no phase separation • pore formation ; mechanical force by cold-stretching	• polymer and diluent • solid-liquid or liquid-liquid phase-separation • pore formation : removal of diluent
Advantage	• relatively easy handling • no solvent and clean process	• relatively easy pore size control
Disadvantage	• difficult pore size control • relatively small pore size	• waste solvent problem • relatively complex process

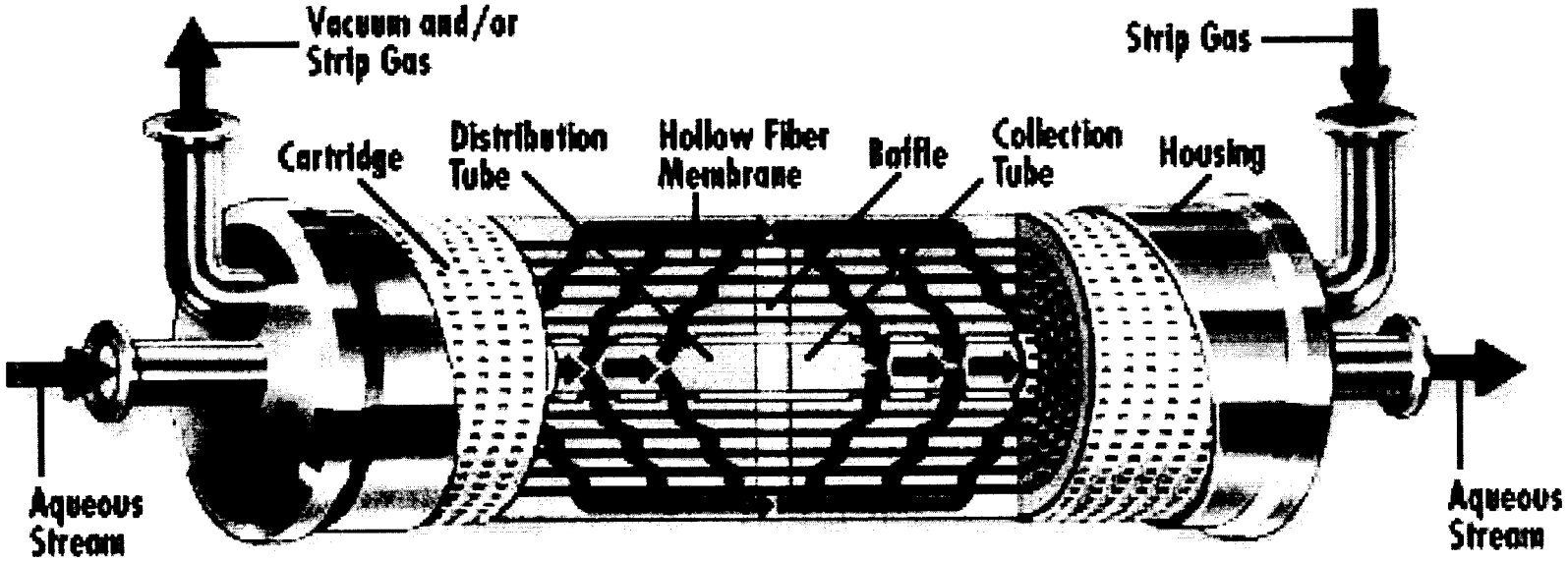
Membranes and Modules for Degassing

Commercial Products

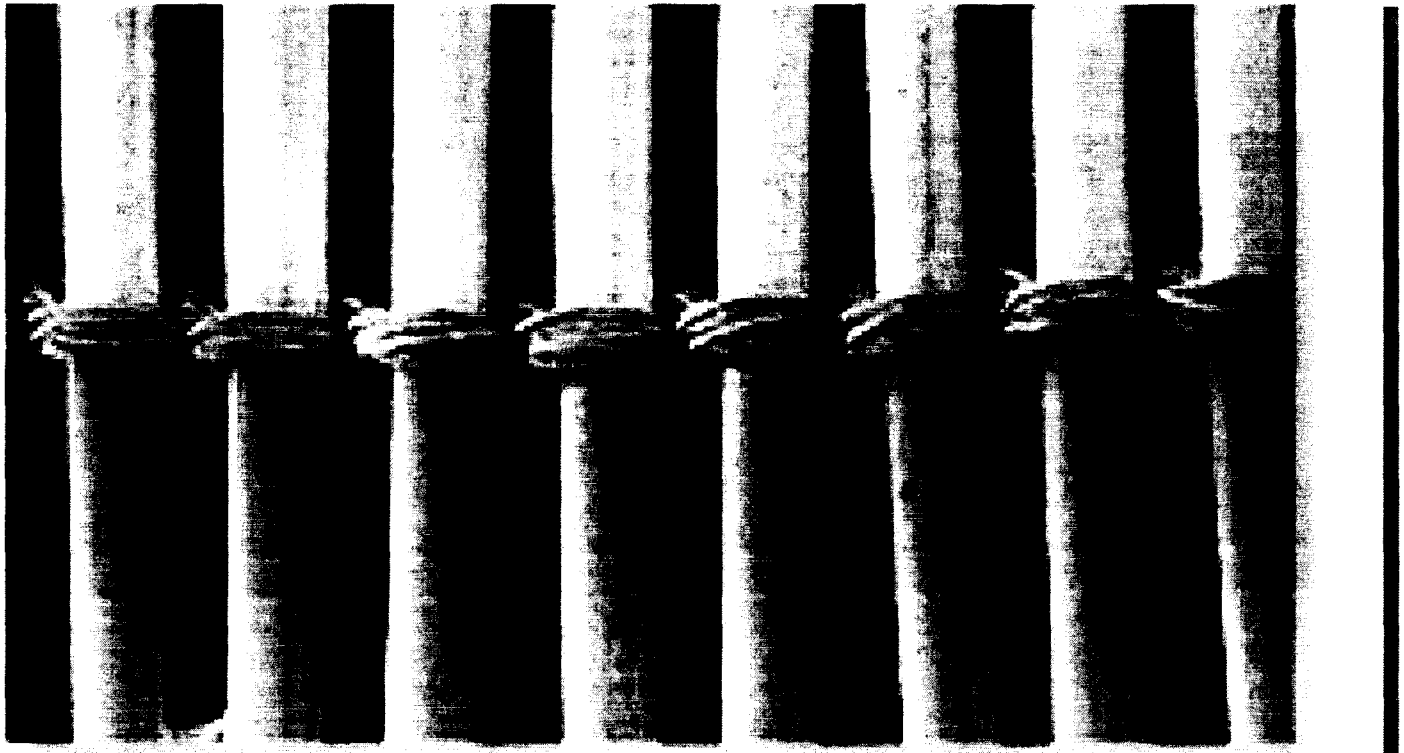
- Celgard : Liqui-Cel ®
- DaiNippon Ink and Chemical : Separel ®
- Ecolochem : Delta Flow ®

product	membrane	housing	Remarks
Liqui-Cel ®	PP	PP, PVDF, St.St.	O_2, CO_2, N_2 Removal
Separel ®	Poly4-methyl Pentene-1	PP	O_2 Removal
Delta Flow ®	PP	N/A	O_2, CO_2 Removal

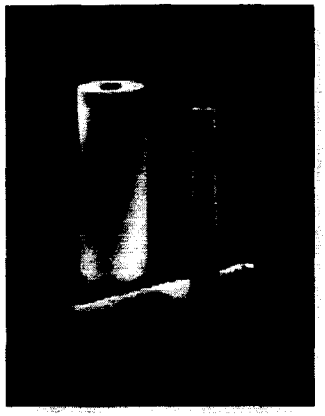
Expoloded View of Degassing Module



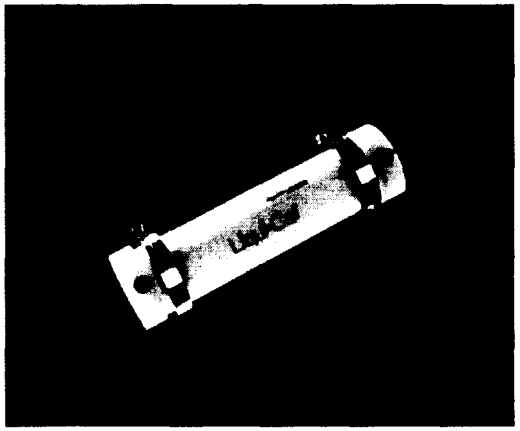
Membrane Array in the Module



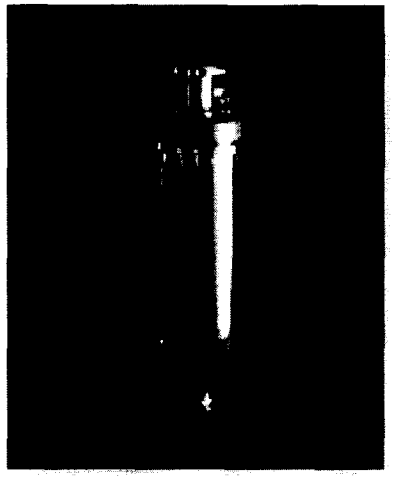
Degassing Module and Vessel



HF module



in Plastic Vessel



in St. St. Vessel

Standardization of Degassing Membrane and Module

- Status
  - Not well established
  - Depends on specifications of company
  - Need growth for standardization for the increasing market
  - ATS, Hansonic Co., Kyung Hee Univ. in Korea

- Items for Standardization
  - Performance test
  - Pressure drop test
  - Integrity test
  - Defect test
  - Data processing

rerformance of Degassing

$$\left[\frac{(C_i - C_0)}{C_i}\right] \times 100$$

$$C_o / C_i = e^{-kaL/vo}$$

$$1/k = 1/k_l + 1/k_m + 1/k_v$$

C_o = *outlet-dissolved-gas concentration*
C_i = *inlet- dissolved-gas concentration*
k = *mass-transfer coefficient*
a = *surface area*
L = *length of membrane*
vo = *velocity of the fluid*
k_l = *local k in liquid phase*
k_l = *local k in membrane*
k_l = *local k in vapor phase*

- The dominant resistance to mass tansfer for oxygen is in the water-phase.

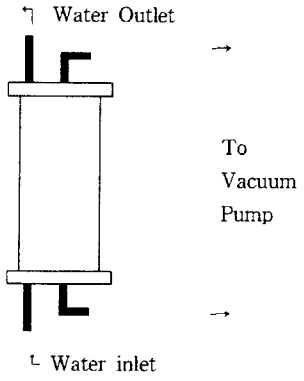
$$\mathbf{k \sim k_l}$$

Mode of Operation

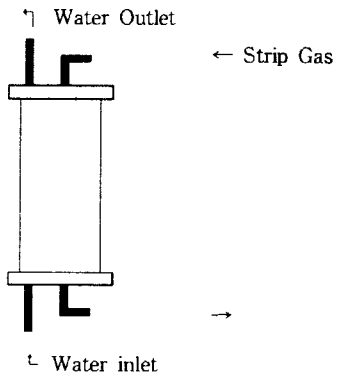
Water flow path mode

- in shell side : popular
- in lumen side : less efficient and high pressure drop

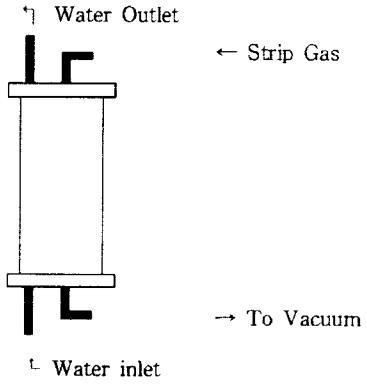
Degassing mode



Vacuum

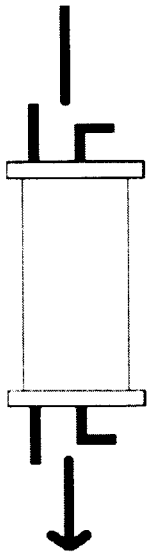


Sweep Gas

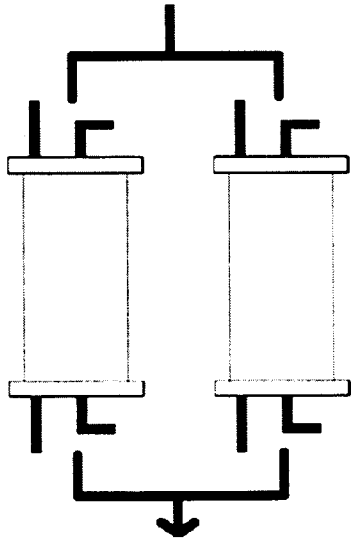


Combo

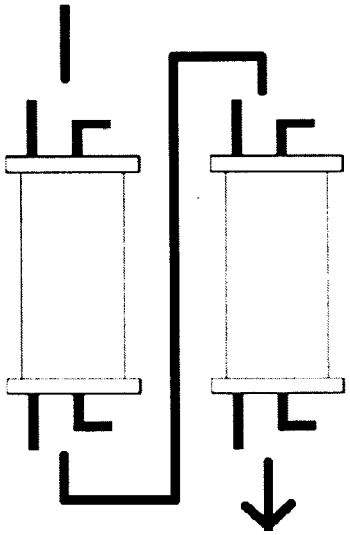
Array of Module



Single



Parallel



Series

Degassing Performance Test

Minimum removal must be specified.

- Liqui-Cel ® : 78 %

Water flow rate : depends on module size

Degassing mode

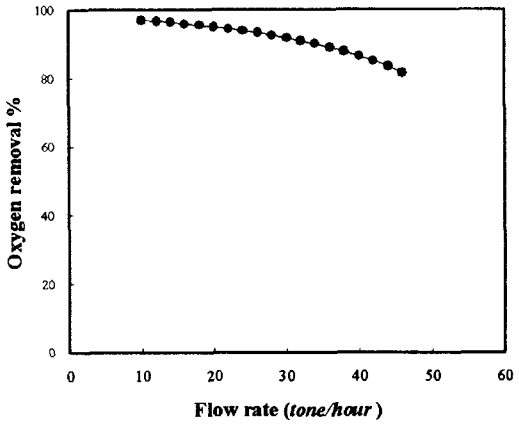
- vacuum pressure
- sweep gas flow rate

Module array mode

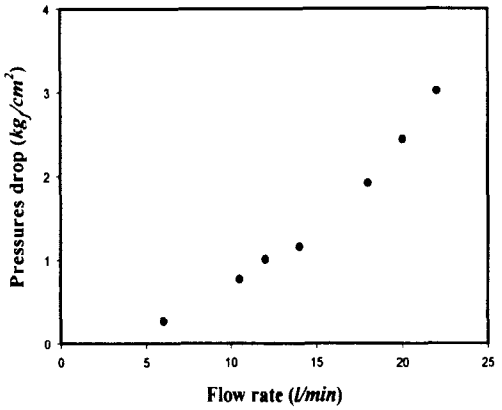
Characteristics of the membrane

- pore size
- thickness
- porosity
- surface property

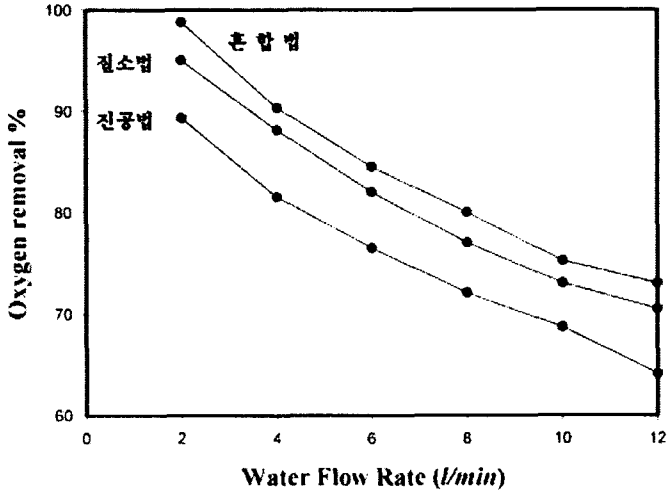
Temperature



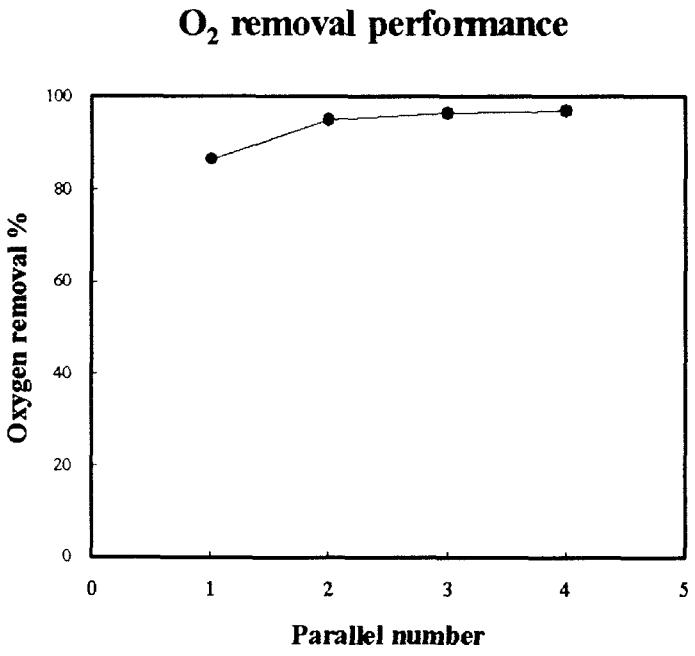
Oxygen Removal



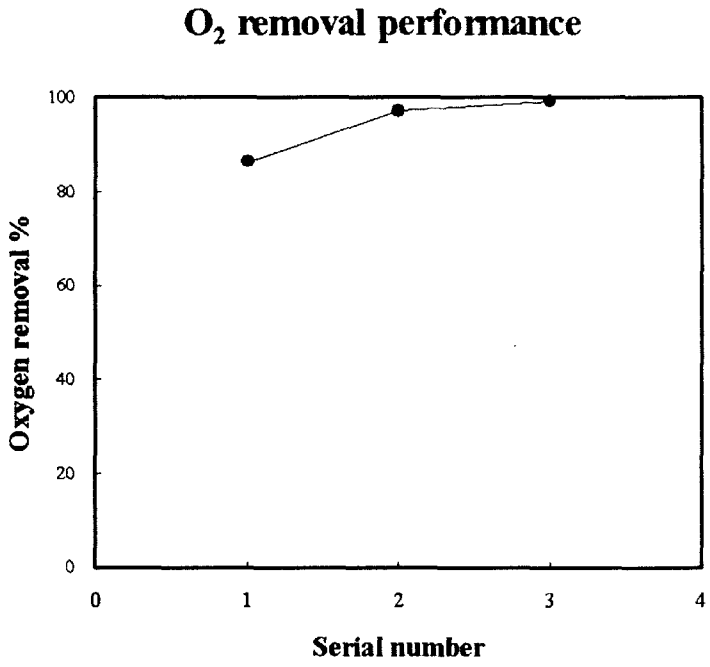
Pressure Drop



Degassing Mode



Parallel Mode



Serial Mode

Pressure Drop Test

Maximum pressure drop is specified.

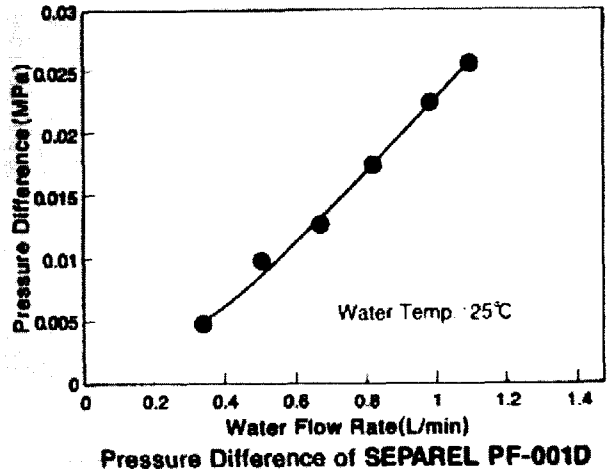
Liqui-Cel ® : 7.7 psi

Water flow rate

Water pressure

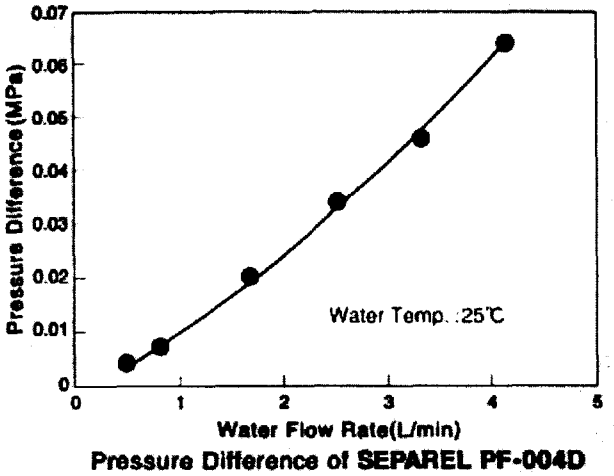
Water flow path mode

Temperature



Pressure Difference of SEPAREL PF-001D

Small module



Pressure Difference of SEPAREL PF-004D

Big module

Integriry Test for Fiber and Seal

- Zero leaks of cartridge in pressurized water
- Water side pressure (60 psi)
- Duration time of test (30 min)
- Pass/Fail

Dimensional Test

- Dimensional change
  - during manufacturing
  - during operation
- Tolerance

Final Inspection

- Visual inspection
- Review and ensure each test result

Future Works

- Establishing test conditions of reproducibility
- Securing equipments needed
- Investigate new applications
- Reflect the requirements of new application
- Correlation with other countries : new trend
- Korean Standard registration
- ISO NWIP registration

Acknowledgements

- Dr. Hyang Sun in ATS
- Celgard Co.
- Hansonic Co.