

5. Filtration





5.1 Filtration Principles



Filtration

■ Filtration

- Separation of particulate or solute components in a fluid suspension or solution
- Separation by size difference
- Flowing under a pressure differential through a porous medium

■ Types

- Conventional or dead-end filtration
 - The fluid flows perpendicular to the medium
 - Results in a cake of solids deposition on the filter medium
 - Used for removal of cells
 - Purification of secreted biomaterials, sterilization
- Crossflow filtration
 - The fluid flows parallel to the medium
 - Minimization of the buildup of a cake
 - Usage
 - Separation of cells
 - Concentration of cells
 - Removal of cell debris after cell lysis
 - Concentration of protein solutions
 - Exchange or removal of salts in a protein solution
 - Removal of viruses

Types of Filtration

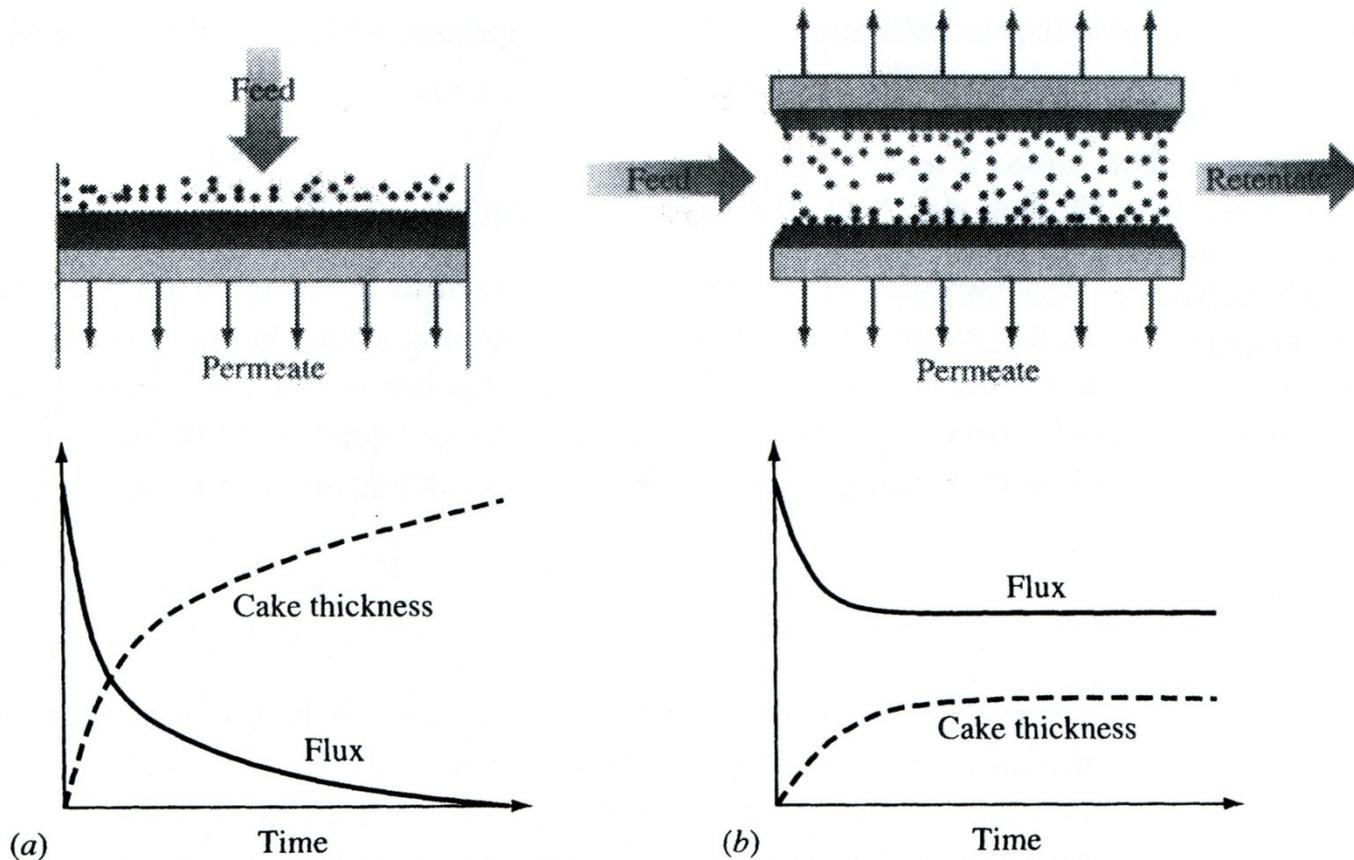


Figure 4.1 Schematic diagrams for (a) dead-end or conventional filtration and (b) crossflow filtration. For dead-end filtration the thickness of the solids buildup increases and the permeate flux decreases with time, ultimately reaching zero. In crossflow filtration the feed can contain either a soluble or a solid solute, which becomes concentrated at the membrane surface; the permeate flux reaches a constant value at steady state.

5.1.1. Conventional Filtration

■ Darcy's law

- $J = 1/A \cdot dV/dt = \Delta p / (\mu_0 R)$ (1)
 - J : transmembrane fluid flux
 - V : volume of filtrate
 - T : time
 - A : cross-sectional area of exposed filter medium
 - Δp : pressure drop through the bed of solids (medium + cake)
 - μ_0 : viscosity of the filtrate
 - R : resistance of the porous bed
- $R = R_m$ (medium) + R_c (cake) (2)
- $R_c = \alpha \rho_c (V/A)$ (3)
 - α : specific cake resistance
 - ρ_c : mass of dry cake solids per volume of filtrate
- $1/A \cdot dV/dt = \Delta p / \{ \mu_0 [\alpha \rho_c (V/A) + R_m] \}$ (1) + (2) + (3) = (4)
- Integration of (4)
- $t/(V/A) = (\mu_0 \alpha \rho_c) / (2\Delta p) \cdot (V/A) + \mu_0 R_m / \Delta p$

Filtration of *Streptomyces* cells

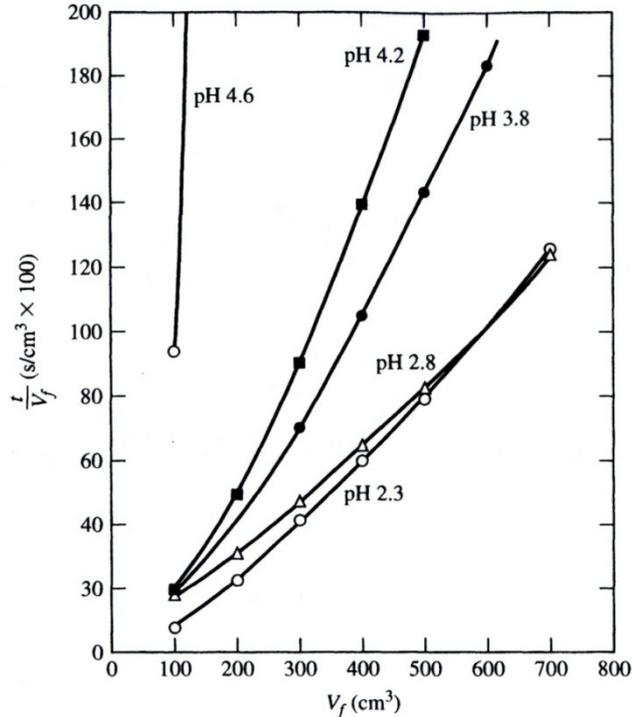


Figure 4.2 Filtration data for *Streptomyces griseus* broth with $\Delta p = 2.0$ bar. The filter medium was of cotton cloth, and diatomaceous earth filter aid was added to the broth. (Data from S. Shirato and S. Esumi, "Filtration of a culture broth of *Streptomyces griseus*" *J. Ferment. Technol. (Japan)*, vol. 41, p. 87, 1963.)

- **$t/(V/A)$ vs. V/A curve**
 - Not perfectly straight
 - Increase in slope with the volume filtered
 - Increase in α as the cake is compressed
- **Specific resistance of cake vs. pressure drop across the cake**
 - $\alpha = \alpha' (\Delta p_c)^s$
 - Δp_c : pressure drop across the cake
 - α' , s : empirical constants
 - S : cake compressibility factor (0 ~ 1)
 - 0: no compression (e.g. sand)

Washing of the Filter Cake

- $R' = (1 - E/100)^n$
 - R' : the weight fraction of solute remaining in the cake after washing
 - $R' = 1.0$ before washing
 - E : the percentage wash efficiency
 - 35% ~ 86%
 - n : the volume of wash liquid (V_w) / volume of liquid in the unwashed cake (V_r)
- $t_w/t_f = 2 V_w/V_r \cdot V_r/V_f = 2nf$
 - V_f : filtrate volume
 - t_f : filtration time
 - t_w : wash time

5.1.2. Crossflow Filtration

■ Classification

- Filtration of soluble components
 - Ultrafiltration membrane
 - Smaller molecules than the cut off size of membrane will pass through the membrane
 - Gel layer: accumulation of proteins on the membrane surface
 - Increase in osmolarity near the membrane surface : solvent gradient against the applied Δp
- Filtration of insoluble components
 - Microfiltration membrane
 - Formation of a cake on the surface

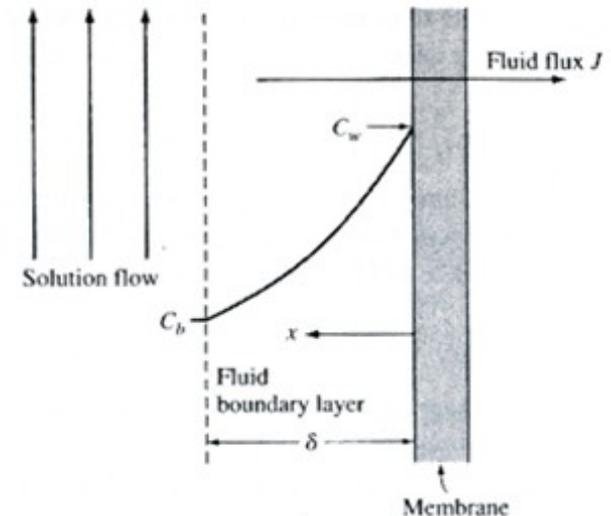
Dissolved Species

■ Concentration polarization

- Higher solute concentration at the membrane surface (c_w) than in the bulk (c_b)

■ At steady state of mass transfer across membrane

- $Jc = -\mathcal{D} dc/dx$
 - J : transmembrane fluid flux
volume/ (length² • time)
 - c : concentration of the solute
 - \mathcal{D} : diffusion coefficient of solute
- $J = \mathcal{D}/\delta \ln (c_w / c_b)$
 - δ : boundary layer thickness
- $c_w / c_b = \exp (J\delta / \mathcal{D}) = \exp (J/k)$
 - \mathcal{D} / δ : mass transfer coefficient k
 - c_w / c_b : polarization modulus
 - high c_w / c_b can cause formation of the solids of gel layer on the membrane surface



Ultrafiltration flux vs. Concentration of solutions

- Estimation of c_w
 - Intercept at zero flux

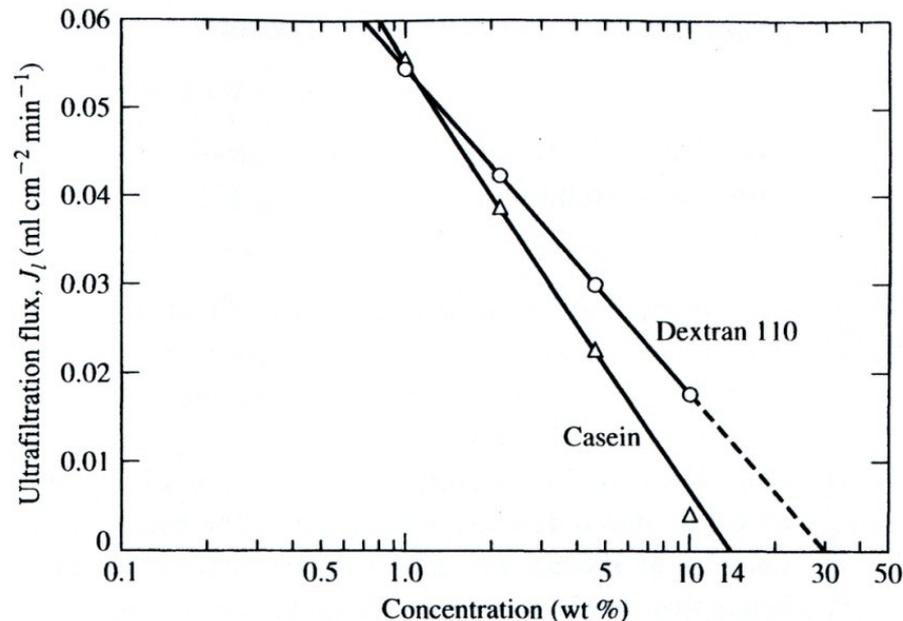


Figure 4.4 Decline in ultrafiltration flux with increasing concentration for solutions of casein and Dextran (MW 110,000). Data were obtained in thin-channel recirculating flow cells. (Data from W. F. Blatt, A. Dravid, A. S. Michaels, and L. Nelsen, "Solute polarization and cake formation in membrane ultrafiltration: Causes, consequences, and control techniques," in *Membrane Science and Technology*, J. E. Flinn, ed., p. 63, Plenum Press, New York, 1970.)

Laminar Flow

■ Leveque or Graetz solutions

- k (mass transfer coefficient) = $0.816 (\gamma_w D^2/L)^{1/3}$
 - γ_w : fluid shear rate at the membrane surface
 - L : length of the flow channel over the membrane
 - 0.816: applicable for the gel-polarized condition where the solute concentration at the wall is constant
- γ_w for a rectangular slit of height $2h$
 - $\gamma_w = 3u_b/h$, u_b : bulk stream velocity
- γ_w for a circular tube of diameter D
 - $\gamma_w = 8u_b/D$

Turbulent flow

■ Empirical correlations for turbulent flow

- Sh (Sherwood number) = $kD_h/\mathcal{D} = f(\text{Re}, \text{Sc}, L/D_h)$
- $k = \mathcal{D} \text{Sh}/D_h$
 - Re (Reynolds number): $D_h u_b \rho / \mu$
 - Turbulent flow when $\text{Re} > 2000$
 - Sc (Schmidt number) : $\mu / (\rho \mathcal{D})$
 - D_h : equivalent diameter of the channel = $4(\text{cross-section area}/\text{wetted perimeter})$
 - ρ : density of fluid
 - μ : viscosity of fluid
- Typical correlation: $\text{Sh} = 0.082 \text{Re}^{0.69} \text{Sc}^{0.33}$

Example : Determination of the polarization modulus c_w / c_b

■ Operation condition of ultrafiltration

- Flow channel tubes
 - Diameter : 0.1 cm, length : 100 cm
- $\mathcal{D} : 9 \times 10^{-7} \text{ cm}^2/\text{s}$
- $\mu : 1.2 \text{ cp (centipoise)} = 0.012\text{g}/(\text{cm s})$
- $\rho : 1.1 \text{ g}/\text{cm}^3$
- $U_b : 300 \text{ cm}/\text{s}$
- Transmembrane flux (J) = $45\text{L m}^{-2} \text{ h}^{-1}$

■ Solution

- $Re = D_h u_b \rho / \mu$
- $Sc = \mu / (\rho \mathcal{D})$
- $k = \mathcal{D} Sh / D_h$
 - $Sh = 0.082 Re^{0.69} Sc^{0.33}$
- $c_w / c_b = \exp (J / k)$

Effect of concentration polarization to fluid flow

■ $J = (\Delta p - \sigma \Delta \pi) / [\mu_0(R_m + R_p)]$

■ Δp : pressure difference between the bulk fluid and the permeate

■ $\Delta \pi$: osmotic pressure

■ σ : reflection coefficient for the solute

1: no passage, 0: free passage

■ For an ideal dilute solution

$$\Delta \pi = RTc_w$$

■ R_p : resistance of the polarized boundary layer, gel layer next to the surface of membrane

■ μ_0 : viscosity of the permeate

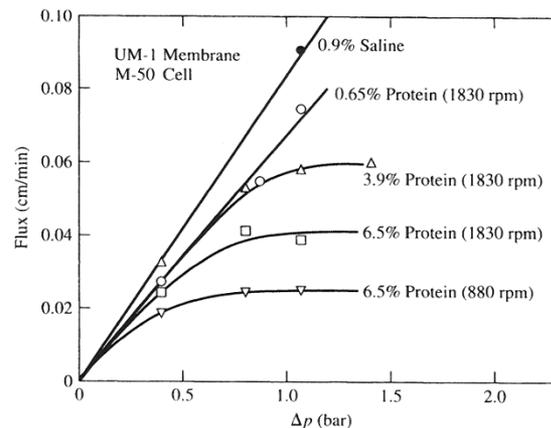


Figure 4.5 Ultrafiltration flux as a function of pressure drop in a stirred cell with varying protein concentration and stirring rate. (Data from E. S. Perry, ed., *Progress in Separation and Purification* vol. 1, p. 318, Wiley, New York, 1968.)

Suspended Particles 1

: 1 to 30~40 μm

- Shear-induced diffusion theory (up to 30~40 μm particles)
 - Zydney and Colton
 - Shear-induced hydrodynamic diffusion of spherical particles
 - $\mathcal{D}_s = 0.3 \gamma_w a^2$ (for $0.2 < \Phi < 0.45$)
 - Φ : particle volume fraction in the bulk suspension
 - a : particle radius
 - γ_w : fluid shear rate at the membrane surface
 - Substitution to $k = 0.816 (\gamma_w \mathcal{D}^2/L)^{1/3}$
 - $k = 0.366 \gamma_w (a^4/L)^{1/3}$
 - Stronger dependence on γ_w than dissolved species

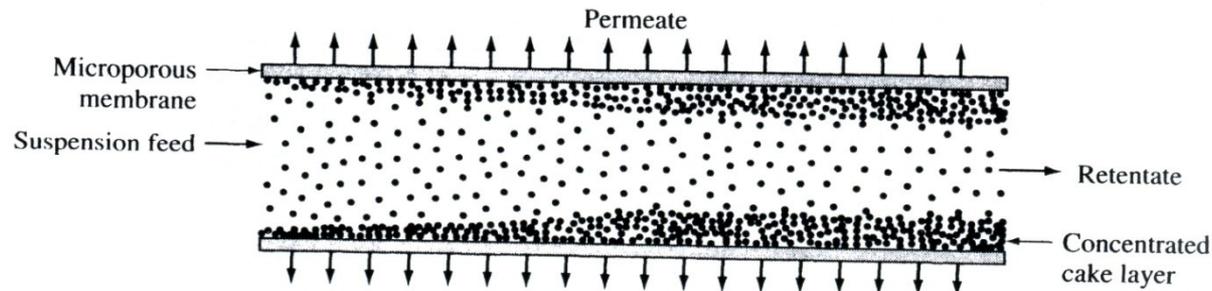


Figure 4.6 Illustration of the buildup of a cake layer at the membrane surface in crossflow filtration with suspended solids in the feed.

Suspended Particles 2

: Inertial lift (larger particles)

- When Reynolds number based on the particle size is not negligible
- For fast laminar flow of dilute suspensions with thin fouling layers
- $J = 0.036\rho_0 a^3 \gamma_w^2 / \mu_0$
 - ρ_0 : density of permeate
 - μ_0 : viscosity of permeate
 - Strong dependence on particle size and shear rate at the membrane surface



5.2 Filter Media and Equipment



Conventional Filtration

■ Filter media

■ In biotechnology

- Woven fabrics : cut off 10 μm
- Metal fabrics or screens : cut off 5 μm
 - Nickel, copper, brass, aluminum, steel, stainless steel etc.
- Rigid porous media
 - Sheets and tubes
 - Sintered stainless steel, silica, porcelain, plastics

■ Sterile filtration

- Membrane filter
 - Cellulose esters, other polymers
 - Pore sizes: 0.22 or 0.45 μm
- Depth filter
 - Compacted beds of pads of fibrous materials (eg. glass wool)
 - High efficiency particulate air (HEPA) filter
 - » Sterile filtration of air
 - » Collect microbes and other airborne particles

Conventional Filtration

■ Filter aids

- Powdered solid improving the filtration operation
- Properties
 - Highly permeable
 - Chemical resistance
 - Compatibility with the product
- Applications
 - Mix with the suspension
 - Less compression, faster filtration
 - Precoat the filter
- Types
 - Diatomite
 - Skeletal remain of diatoms (algae)
 - Mostly silica
 - Perlite
 - A glassy volcanic material
 - Aluminum silicate with some combined water
 - Can be used for rough filtration

Conventional Filtration

■ Equipment

■ Classification

- Driving force: pressure, vacuum, gravity
- Operation: batch, semicontinuous, continuous

■ Types

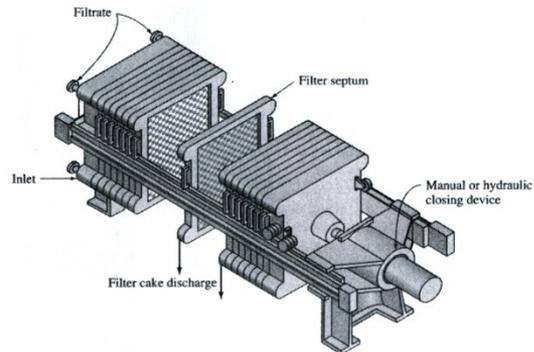


Figure 4.7 Plate-and-frame filter press.

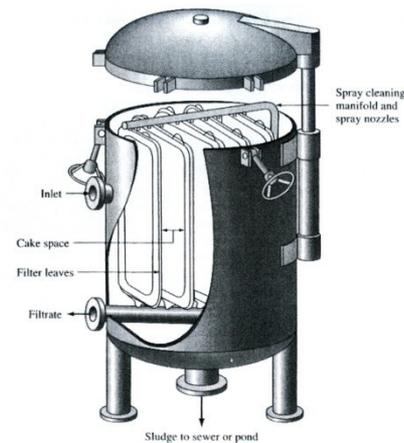


Figure 4.8 Vertical leaf filter.

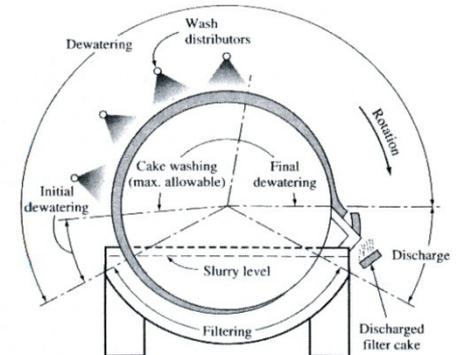


Figure 4.9 Rotary vacuum filter.

Crossflow Filtration

■ Filter media (membranes)

- Ultrafiltration (UF)
 - Pore size: 0.001 to 0.1 μm
 - Molecular weight cutoff: 90% rejection
 - MWCO 1kDa to 1,000 kDa
- Microfiltration (MF)
- Reverse osmosis (RO) or hyperfiltration membranes
 - Pass only water and a very low flux of solutes

■ Membrane structure

- Homogenous
 - Homogenous pore size
- Asymmetric
 - Thin layer with small pores and thicker layer with larger pores
- Composite
 - Asymmetric membrane consisting of two different types of material

■ Materials

- Polymers
 - Cellulose acetate, polyamide, polyether, polycarbonate, polyester, polypropylene, polyethylene, poly(vinylidene fluoride) (PVDF) etc.
- Inorganic materials
 - Ceramics, zirconium oxide, borosilicate glass, stainless steel, silver

Crossflow Filtration

■ Equipment

■ Module

- Physical unit containing membranes

- Requirements

- Mechanical:

- » Obtain effective separation of the feed and permeate streams
- » Physical support for the membrane

- Hydrodynamic:

- » Minimize pressure drops through the module
- » Optimize solute mass transfer
- » Minimize particulate plugging or fouling

- Economic

- » Maximize membrane packing density
- » Minimize manufacturing costs
- » Easy access for cleaning and membrane replacement
- » Sufficient chemical resistance and operation lifetime
- » Incorporate modularity of design for easy scale up, staging, or cascading

Crossflow Filtration

- Types of module
 - Hollow fiber
 - Highly resistant to plugging
 - Can be cleaned by backflushing
 - Flat plate systems
 - Spiral-wound modules
 - Susceptible to fouling
 - Rotating cylinder device
 - Rate of mass transfer is determined mainly by the rate of rotation of the inner cylinder (> 300 rpm)
 - High energy demand

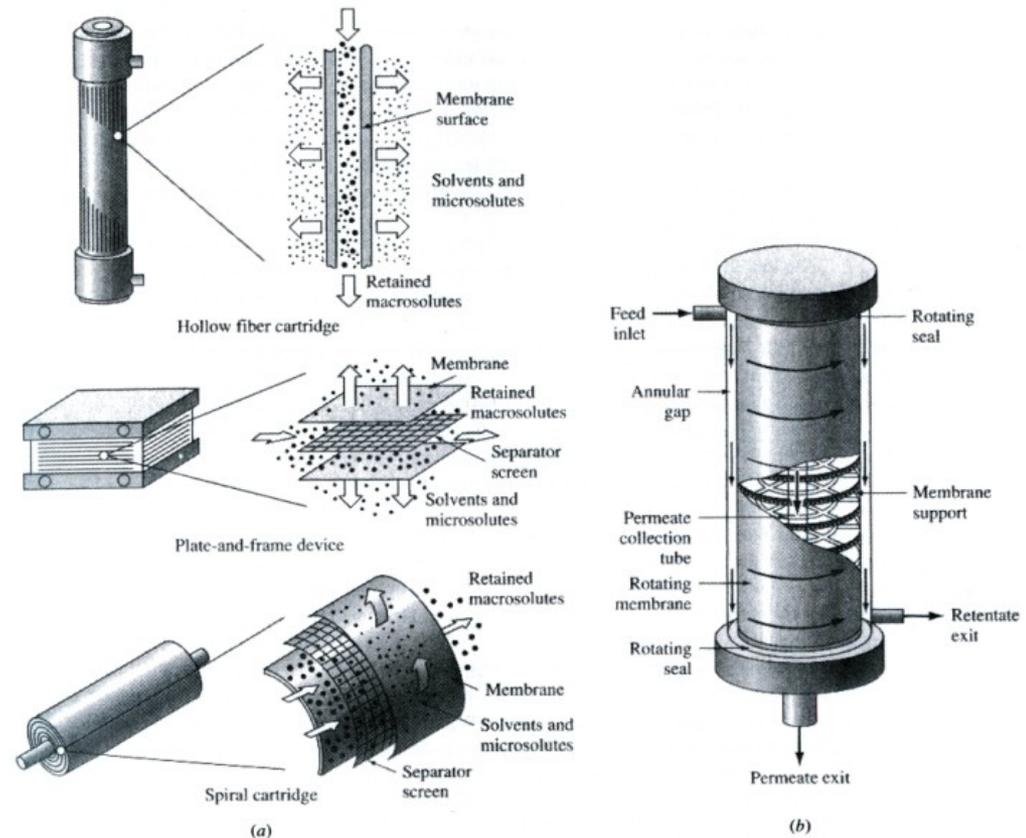


Figure 4.11 Schematic representations of filter modules. (a) Hollow fiber, plate, and spiral-wound membrane modules. (b) A rotating cylinder module.

Membrane Fouling

- **Result from**
 - Physical and/or chemical interactions between the membrane and various components in the process stream
- **Result in**
 - A decline in the permeate flux
 - A change in the membrane selectivity
- **Proteins are the main contributor to fouling**
 - Protein adsorption to the membrane
 - Can be minimized by using hydrophilic membrane or reducing pore size
 - Protein deposition to the adsorbed protein
 - Protein accumulation by concentration polarization → formation of gel layer
 - Can be minimized by increasing the fluid shear rate

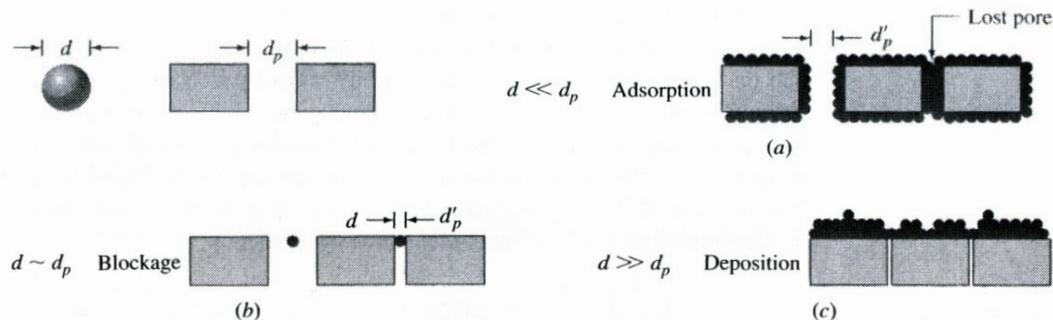


Figure 4.12 Mechanisms of membrane fouling. (a) Pore narrowing and constriction. (b) Pore plugging. (c) Solute deposition and formation of a gel or cake layer. Dimensions: d , protein or particle diameter, d_p , clean pore diameter, d'_p , effective pore diameter in the presence of adsorbed proteins or particles.