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SCHOOL OF BIO AND CHEMICAL ENGINEERING

DEPARTMENT OF CHEMICAL ENGINEERING

UNIT – I – THERMAL SEPERATION SCHA3005

Introduction to mass transfer

When a system contains two or more components whose concentrations vary from point to point, there is a natural tendency for mass to be transferred, minimizing the concentration differences within a system. The transport of one constituent from a region of higher concentration to that of a lower concentration is called mass transfer.

The transfer of mass within a fluid mixture or across a phase boundary is a process that plays a major role in many industrial processes. Examples of such processes are:

(i) Dispersion of gases from stacks

(ii) Removal of pollutants from plant discharge streams by absorption

(iii) Stripping of gases from wastewater

(iv) Neutron diffusion within nuclear reactors

(v) Air conditioning

Many of air day-by-day experiences also involve mass transfer, for example:

(i) A lump of sugar added to a cup of coffee eventually dissolves and then eventually diffuses to make the concentration uniform.
(ii) Water evaporates from ponds to increase the humidity of passing-air-stream
(iii) Perfumes presents a pleasant fragrance which is imparted throughout the surrounding atmosphere.

The mechanism of mass transfer involves both molecular diffusion and convection.

Properties of mixtures

Mass transfer always involves mixtures. Consequently, we must account for the variation of physical properties which normally exist in a given system. When a system contains three or more components, as many industrial fluid streams do, the problem becomes unwise very quickly. The conventional engineering approach to problems of multicomponent systems is to attempt to reduce them to representative binary (i.e., two-component) systems.

Concentration of species

Concentration of species in multicomponent mixture can be expressed in many ways. For species A, mass concentration denoted by ρ_A is defined as the mass of A,m_A per unit volume of the mixture.

$$\rho_A = \frac{m_A}{V} \qquad \text{----------} \quad (1)$$

The total mass concentration density ρ is the sum of the total mass of the mixture in unit volume:

$$\rho = \sum_i \rho_i$$

where ρ_i is the concentration of species i in the mixture.

Molar concentration of, A, C_A is defined as the number of moles of A present per unit volume of the mixture.

By definition,

$$Number\ of\ moles = \frac{mass\ of\ A}{molecular\ weight\ of\ A}$$

$$n_A = \frac{m_A}{M_A} \quad \text{---------- (2)}$$

Therefore from (1) & (2)

$$C_A = \frac{n_A}{V} = \frac{\rho_A}{M_A}$$

For ideal gas mixtures,

$$n_A = \frac{p_A V}{RT} \quad \text{[from Ideal gas law PV = nRT]}$$

$$C_A = \frac{n_A}{V} = \frac{p_A}{RT}$$

where p_A is the partial pressure of species A in the mixture. V is the volume of gas, T is the absolute temperature, and R is the universal gas constant.

The total molar concentration or molar density of the mixture is given by

$$C = \sum_i C_i$$

Velocities

In a multicomponent system the various species will normally move at different velocities; and evaluation of velocity of mixture requires the averaging of the velocities of each species present.

If v_I is the velocity of species i with respect to stationary fixed coordinates, then mass-average velocity for a multicomponent mixture defined in terms of mass concentration is,

$$v = \frac{\sum_i \rho_i v_i}{\sum_i \rho_i} = \frac{\sum_i \rho_i v_i}{\rho}$$

By similar way, molar-average velocity of the mixture v^* is

$$\nu^* = \frac{\sum_i C_i V_i}{C}$$

For most engineering problems, there will be title difference in ν^* and ν and so the mass average velocity, ν, will be used in all further discussions.

The velocity of a particular species relative to the mass-average or molar average velocity is termed as diffusion velocity

(i.e.) Diffusion velocity = $\nu_i - \nu$

The mole fraction for liquid and solid mixture, x_A ,and for gaseous mixtures, y_A, are the molar concentration of species A divided by the molar density of the mixtures.

$$x_A = \frac{C_A}{C} \quad \text{(liquids and solids)}$$

$$y_A = \frac{C_A}{C} \quad \text{(gases).}$$

The sum of the mole fractions, by definition must equal 1;

(i.e.) $$\sum_i x_i = 1$$

$$\sum_i y_i = 1$$

by similar way, mass fraction of A in mixture is;

$$w_A = \frac{\rho_A}{\rho}$$

The molar composition of a gas mixture at 273 K and $1.5 * 10^5$ Pa is:

O_2	7%
CO	10%
CO_2	15%
N_2	68%

Determine

a) the composition in weight percent
b) average molecular weight of the gas mixture
c) density of gas mixture
d) partial pressure of O_2.

Calculations:
Let the gas mixture constitutes 1 mole. Then

O_2 = 0.07 mol
CO = 0.10 mol
CO_2 = 0.15 mol
N_2 = 0.68 mol

Molecular weight of the constituents are:

O_2 = 2 * 16 = 32 g/mol
CO = 12 + 16 = 28 g/mol
CO_2 = 12 + 2 * 16 = 44 g/mol
N_2 = 2 * 14 = 28 g/mol

Weight of the constituents are: (1 mol of gas mixture)

O_2 = 0.07 * 32 = 2.24 g
CO = 0.10 * 28 = 2.80 g
CO_2 = 0.15 * 44 = 6.60 g
N_2 = 0.68 * 28 = 19.04 g

Total weight of gas mixture = 2.24 + 2.80 + 6.60 + 19.04
= 30.68 g

Composition in weight percent:

$$O_2 = \frac{2.24}{30.68} * 100 = 7.30\%$$

$$CO = \frac{2.80}{30.68} * 100 = 9.13\%$$

$$CO_2 = \frac{6.60}{30.68} * 100 = 21.51\%$$

$$N_2 = \frac{19.04}{30.68} * 100 = 62.06\%$$

Average molecular weight of the gas mixture $M = \frac{\text{Weight of gas mixture}}{\text{Number of moles}}$

$$M = \frac{30.68}{1} = 30.68\ g/mol$$

Assuming that the gas obeys ideal gas law,
PV = nRT

$$\frac{n}{V} = \frac{P}{RT}$$

$$\frac{n}{V} = \text{molar density} = \rho_m$$

Therefore, density (or mass density) = $\rho_m M$
Where M is the molecular weight of the gas.

$$Density = \rho_m M = \frac{PM}{RT} = \frac{1.5 * 10^5 * 30.68}{8314 * 273} \; kg/m^3$$

$= 2.03 \text{ kg/m}^3$

pressure of O_2 = [mole fraction of O_2] * total pressure

$$= \frac{7}{100} * \left(1.5 * 10^5\right)$$

$= 0.07 * 1.5 * 10^5$

$= 0.105 * 10^5$ Pa

Diffusion flux

Just as momentum and energy (heat) transfer have two mechanisms for transport-molecular and convective, so does mass transfer. However, there are convective fluxes in mass transfer, even on a molecular level. The reason for this is that in mass transfer, whenever there is a driving force, there is always a net movement of the mass of a particular species which results in a bulk motion of molecules. Of course, there can also be convective mass transport due to macroscopic fluid motion. In this chapter the focus is on molecular mass transfer.

The mass (or molar) flux of a given species is a vector quantity denoting the amount of the particular species, in either mass or molar units, that passes per given increment of time through a unit area normal to the vector. The flux of species defined with reference to fixed spatial coordinates, N_A is

$$N_A = C_A v_A \qquad \text{---------- (1)}$$

This could be written interms of diffusion velocity of A, (i.e., $v_A - v$) and average velocity of the mixture, v, as

$$N_A = C_A (v_A - v) + C_A v \qquad \text{---------- (2)}$$

By definition

$$v = v^* = \frac{\sum_i C_i v_i}{C}$$

Therefore, equation (2) becomes

$$N_A = C_A (v_A - v) + \frac{C_A}{C} \sum_i C_i v_i$$

$$= C_A (v_A - v) + y_A \sum_i C_i v_i$$

For systems containing two components A and B,

$$N_A = C_A (v_A - v) + y_A (C_A v_A + C_B v_B)$$

$$= C_A (v_A - v) + y_A (N_A + N_B)$$

$$N_A = C_A (v_A - v) + y_A N \quad \text{---------- (3)}$$

The first term on the right hand side of this equation is diffusional molar flux of A, and the second term is flux due to bulk motion.

Fick's law:

An empirical relation for the diffusional molar flux, first postulated by Fick and, accordingly, often referred to as Fick's first law, defines the diffusion of component A in an isothermal, isobaric system. For diffusion in only the Z direction, the Fick's rate equation is

$$J_A = -D_{AB} \frac{dC_A}{dZ}$$

where D_{AB} is diffusivity or diffusion coefficient for component A diffusing through component B, and dC_A / dZ is the concentration gradient in the Z-direction.

A more general flux relation which is not restricted to isothermal, isobasic system could be written as

$$J_A = -C D_{AB} \frac{dy_A}{dZ} \quad \text{---------- (4)}$$

using this expression, Equation (3) could be written as

$$N_A = -C D_{AB} \frac{dy_A}{dZ} + y_A N \quad \text{---------- (5)}$$

Relation among molar fluxes:

For a binary system containing A and B, from Equation (5),

$$N_A = J_A + y_A N$$

or $$J_A = N_A + y_A N \quad \text{---------- (6)}$$

Similarly,

$$J_B = N_B + y_B N \quad \text{---------- (7)}$$

Addition of Equation (6) & (7) gives,

$$J_A + J_B = N_A + N_B - (y_A + y_B) N \quad \text{---------- (8)}$$

By definition $N = N_A + N_B$ and $y_A + y_B = 1$.
Therefore equation (8) becomes,

$$J_A + J_B = 0$$

$$J_A = -J_B$$

$$C D_{AB} \frac{d y_A}{d z} = - C D_{BA} \frac{d y_B}{d Z} \quad \text{---------- (9)}$$

From $y_A + y_B = 1$
$dy_A = - dy_B$

Therefore Equation (9) becomes,

$$D_{AB} = D_{BA} \quad \text{---------- (10)}$$

This leads to the conclusion that diffusivity of A in B is equal to diffusivity of B in A.

Diffusivity

Fick's law proportionality, D_{AB}, is known as mass diffusivity (simply as diffusivity) or as the diffusion coefficient. D_{AB} has the dimension of L^2 / t, identical to the fundamental dimensions of the other transport properties: Kinematic viscosity, $\nu\eta = (\mu / \rho)$ in momentum transfer, and thermal diffusivity, α ($= k / \rho C_\rho$) in heat transfer.

Diffusivity is normally reported in cm^2 / sec; the SI unit being m^2 / sec.

Diffusivity depends on pressure, temperature, and composition of the system.

In table, some values of D_{AB} are given for a few gas, liquid, and solid systems.

Diffusivities of gases at low density are almost composition independent, increase with the temperature and vary inversely with pressure. Liquid and solid diffusivities are strongly concentration dependent and increase with temperature.

General range of values of diffusivity:

Gases :	5×10^{-6}	----------	1×10^{-5}	m^2 / sec.
Liquids :	10^{-6}	----------	10^{-9}	m^2 / sec.
Solids :	5×10^{-14}	----------	1×10^{-10}	m^2 / sec.

In the absence of experimental data, semi theoretical expressions have been developed which give approximation, sometimes as valid as experimental values, due to the difficulties encountered in experimental measurements.

Diffusivity in Gases:

Pressure dependence of diffusivity is given by

$$D_{AB} \propto \frac{1}{p}$$ (for moderate ranges of pressures, upto 25 atm).

And temperature dependency is according to

$$D_{AB} \propto T^{3/2}$$

Diffusivity of a component in a mixture of components can be calculated using the diffusivities

for the various binary pairs involved in the mixture. The relation given by Wilke is

$$D_{1-mixture} = \frac{1}{\frac{y'_2}{D_{1-2}} + \frac{y'_3}{D_{1-3}} + \ldots\ldots\ldots + \frac{y'_n}{D_{1-n}}}$$

Where $D_{1\text{-mixture}}$ is the diffusivity for component 1 in the gas mixture; $D_{1\text{-n}}$ is the diffusivity for the binary pair, component 1 diffusing through component n; and y'_n is the mole fraction of component n in the gas mixture evaluated on a component –1 – free basis, that is

$$y'_2 = \frac{y_2}{y_2 + y_3 + \ldots\ldots y_n}$$

Determine the diffusivity of Co_2 (1), O_2 (2) and N_2 (3) in a gas mixture having the composition:

Co_2 : 28.5 %, O_2 : 15%, N_2 : 56.5%,

The gas mixture is at 273 k and $1.2 * 10^5$ Pa. The binary diffusivity values are given as: (at 273 K)

$D_{12} P = 1.874\ m^2$ Pa/sec
$D_{13} P = 1.945\ m^2$ Pa/sec
$D_{23} P = 1.834\ m^2$ Pa/sec

Calculations:

Diffusivity of Co_2 in mixture

$$D_{1m} = \frac{1}{\frac{y_2'}{D_{12}} + \frac{y_3'}{D_{13}}}$$

where $y_2' = \frac{y_2}{y_2 + y_3} = \frac{0.15}{0.15 + 0.565} = 0.21$

$$y_3' = \frac{y_3}{y_2 + y_3} = \frac{0.565}{0.15 + 0.565} = 0.79$$

Therefore $D_{1m} P = \frac{1}{\frac{0.21}{1.874} + \frac{0.79}{1.945}}$

$= 1.93\ m^2$.Pa/sec

Since $P = 1.2 * 10^5$ Pa,

$$D_{1m} = \frac{1.93}{1.2 * 10^5} = 1.61 * 10^{-5}\ m^2/\text{sec}$$

Diffusivity of O_2 in the mixture,

$$D_{2m} = \frac{1}{\frac{y_1'}{D_{21}} + \frac{y_3'}{D_{23}}}$$

Where $y_1' = \frac{y_1}{y_1 + y_3} = \frac{0.285}{0.285 + 0.565} = 0.335$

(mole fraction on-2 free bans).

and

$$y_3' = \frac{y_3}{y_1 + y_3} = \frac{0.565}{0.285 + 0.565} = 0.665$$

and

$D_{21} P = D_{12} P = 1.874 \text{ m}^2\text{.Pa/sec}$

Therefore

$$D_{2m} P = \frac{1}{\frac{0.335}{1.874} + \frac{0.665}{1.834}}$$

$= 1.847 \text{ m}^2\text{.Pa/sec}$

$$D_{2}m = \frac{1.847}{1.2 * 10^5} = 1.539 * 10^{-5} \ m^2/\text{sec}$$

In chemistry, diffusivity limits the rate of acid-base reactions; in the chemical industry, diffusion is responsible for the rates of liquid-liquid extraction. Diffusion in liquids is important because it is slow.

Certain molecules diffuse as molecules, while others which are designated as electrolytes ionize in solutions and diffuse as ions. For example, sodium chloride (NaCl), diffuses in water as ions Na^+ and Cl^-. Though each ions has a different mobility, the electrical neutrality of the solution indicates the ions must diffuse at the same rate; accordingly, it is possible to speak of a diffusion coefficient for molecular electrolytes such as NaCl. However, if several ions are present, the diffusion rates of the individual cations and anions must be considered, and molecular diffusion coefficients have no meaning.

Diffusivity varies inversely with viscosity when the ratio of solute to solvent ratio exceeds five. extremely high viscosity materials, diffusion becomes independent of viscosity.

Diffusivity in solids:

Typical values for diffusivity in solids are shown in table. One outstanding characteristic of these values is their small size, usually thousands of time less than those in a liquid, which are inturn 10,000 times less than those in a gas.

Diffusion plays a major role in catalysis and is important to the chemical engineer. For metallurgists, diffusion of atoms within the solids is of more importance.

Steady State Diffusion

In this section, steady-state molecular mass transfer through simple systems in which the concentration and molar flux are functions of a single space coordinate will be considered.

In a binary system, containing A and B, this molar flux in the direction of z, as given by Eqn (5) is [section 3.3.1]

$$N_A = -C D_{AB} \frac{dy_A}{dz} + y_A (N_A + N_B) \quad \text{--- (1)}$$

3.5.1 Diffusion through a stagnant gas film

The diffusivity or diffusion coefficient for a gas can be measured, experimentally using Arnold diffusion cell. This cell is illustrated schematically in .

The narrow tube of uniform cross section which is partially filled with pure liquid A, is maintained at a constant temperature and pressure. Gas B which flows across the open end of the tub, has a negligible solubility in liquid A, and is also chemically inert to A. (i.e. no reaction between A & B).

Component A vaporizes and diffuses into the gas phase; the rate of vaporization may be physically measured and may also be mathematically expressed interms of the molar flux.

Consider the control volume S Δ z, where S is the cross-sectional area of the tube. Mass balance on A over this control volume for a steady-state operation yields

[Moles of A leaving at z + Δz] – [Moles of A entering at z] = 0.

(i.e.) $$S N_A \Big|_{z+\Delta z} - S N_A \Big|_z = 0. \quad \text{---------- (1)}$$

Dividing through by the volume, SΔZ, and evaluating in the limit as ΔZ approaches zero, we obtain the differential equation

$$\frac{dN_A}{dz} = 0 \quad \text{---------- (2)}$$

This relation stipulates a constant molar flux of A throughout the gas phase from Z_1 to Z_2.

A similar differential equation could also be written for component B as,

$$\frac{dN_B}{dZ} = 0,$$

and accordingly, the molar flux of B is also constant over the entire diffusion path from z_1 and z_2.

Considering only at plane z_1, and since the gas B is insoluble is liquid A, we realize that N_B, the net flux of B, is zero throughout the diffusion path; accordingly B is a stagnant gas.

From equation (1) (of section 3.5)

$$N_A = -C D_{AB} \frac{d y_A}{d z} + y_A (N_A + N_B)$$

Since $N_B = 0$,

$$N_A = -C D_{AB} \frac{d y_A}{d z} + y_A N_A$$

Rearranging,

$$N_A = \frac{-C D_{AB}}{1 - y_A} \frac{d y_A}{d z} \qquad \text{---------- (3)}$$

This equation may be integrated between the two boundary conditions:

at $z = z_1$ $\quad Y_A = Y_{A1}$

And at $z = z_2$ $\quad Y_A = y_{A2}$

Assuming the diffusivity is to be independent of concentration, and realizing that N_A is constant along the diffusion path, by integrating equation (3) we obtain

$$N_A \int_{Z_1}^{Z_2} d z = C D_{AB} \int_{y_{A1}}^{y_{A2}} \frac{-d y_A}{1 - y_A}$$

$$N_A = \frac{C D_{AB}}{Z_2 - Z_1} \ln\left(\frac{1 - y_{A2}}{1 - y_{A1}}\right) \qquad \text{----------(4)}$$

The log mean average concentration of component B is defined as

$$y_{B,lm} = \frac{y_{B2} - y_{B1}}{\ln\left(y_{B2} / y_{B1}\right)}$$

Since $y_B = 1 - y_A$,

$$y_{B,lm} = \frac{(1 - y_{A2}) - (1 - y_{A1})}{\ln\left(y_{A2} / y_{A1}\right)} = \frac{y_{A1} - y_{A2}}{\ln\left(y_{A2} / y_{A1}\right)} \qquad \text{------- (5)}$$

Substituting from Eqn (5) in Eqn (4),

$$N_A = \frac{C D_{AB}}{Z_2 - z_1} \frac{(y_{A1} - y_{A2})}{y_{B,lm}} \quad \text{---------- (6)}$$

For an ideal gas $C = \frac{n}{V} = \frac{p}{RT}$, and

for mixture of ideal gases $y_A = \frac{p_A}{P}$

Therefore, for an ideal gas mixture equation. (6) becomes

$$N_A = \frac{D_{AB}}{RT(z_2 - z_1)} \frac{(p_{A1} - p_{A2})}{p_{B,lm}}$$

This is the equation of molar flux for steady state diffusion of one gas through a second stagnant gas.

Many mass-transfer operations involve the diffusion of one gas component through another non-diffusing component; absorption and humidification are typical operations defined by this equation.

Oxygen is diffusing in a mixture of oxygen-nitrogen at 1 std atm, 25°C. Concentration of oxygen at planes 2 mm apart are 10 and 20 volume % respectively. Nitrogen is non-diffusing.

(a) Derive the appropriate expression to calculate the flux oxygen. Define units of each term clearly.
(b) Calculate the flux of oxygen. Diffusivity of oxygen in nitrogen = $1.89 * 10^{-5}$ m^2/sec.

Solution:

Let us denote oxygen as A and nitrogen as B. Flux of A (i.e.) N_A is made up of two components, namely that resulting from the bulk motion of A (i.e.), Nx_A and that resulting from molecular diffusion J_A:

$$N_A = Nx_A + J_A \quad \text{---------- (1)}$$

From Fick's law of diffusion,

$$J_A = -D_{AB} \frac{dC_A}{dz} \quad \text{---------- (2)}$$

Substituting this equation (1)

$$N_A = Nx_A - D_{AB} \frac{dC_A}{dz} \quad \text{---------- (3)}$$

Since $N = N_A + N_B$ and $x_A = C_A / C$ equation (3) becomes

$$N_A = \left(N_A + N_B\right)\frac{C_A}{C} - D_{AB}\frac{dC_A}{dz}$$

Rearranging the terms and integrating between the planes between 1 and 2,

$$\int \frac{dz}{cD_{AB}} = -\int_{C_{A1}}^{C_{A2}} \frac{dC_A}{N_A C - C_A\left(N_A + N_B\right)} \quad \text{---------- (4)}$$

Since B is non diffusing $N_B = 0$. Also, the total concentration C remains constant. Therefore, equation (4) becomes

$$\frac{z}{CD_{AB}} = -\int_{C_{A1}}^{C_{A2}} \frac{dC_A}{N_A C - N_A C_A}$$

$$= \frac{1}{N_A} \ln \frac{C - C_{A2}}{C - C_{A1}}$$

Therefore,

$$N_A = \frac{CD_{AB}}{z} \ln \frac{C - C_{A2}}{C - C_{A1}} \quad \text{---------- (5)}$$

Replacing concentration in terms of pressures using Ideal gas law, equation (5) becomes

$$N_A = \frac{D_{AB} P_t}{RTz} \ln \frac{P_t - P_{A2}}{P_t - P_{A1}} \quad \text{---------- (6)}$$

where
D_{AB} = molecular diffusivity of A in B
P_T = total pressure of system
R = universal gas constant
T = temperature of system in absolute scale
z = distance between two planes across the direction of diffusion
P_{A1} = partial pressure of A at plane 1, and
P_{A2} = partial pressure of A at plane 2

Given:

$D_{AB} = 1.89 * 10^{-5}$ m^2/sec
P_t = 1 atm = $1.01325 * 10^5$ N/m^2
T = 25°C = 273 + 25 = 298 K
z = 2 mm = 0.002 m
$P_{A1} = 0.2 * 1 = 0.2$ atm (From Ideal gas law and additive pressure rule)
$P_{A2} = 0.1 * 1 = 0.1$ atm

Substituting these in equation (6)

$$N_A = \frac{(1.89 * 10^{-5})(1.01325 * 10^5)}{(8314)(298)(0.002)} \ln\left(\frac{1-0.1}{1-0.2}\right)$$

$= 4.55 * 10^{-5}$ kmol/m^2.sec

Pseudo steady state diffusion through a stagnant film:

In many mass transfer operations, one of the boundaries may move with time. If the length of the diffusion path changes a small amount over a long period of time, a pseudo steady state diffusion model may be used. When this condition exists, the equation of steady state diffusion through stagnant gas' can be used to find the flux.

If the difference in the level of liquid A over the time interval considered is only a small fraction of the total diffusion path, and $t_0 - t$ is relatively long period of time, at any given instant in that period, the molar flux in the gas phase may be evaluated by

$$N_A = \frac{C D_{AB} (y_{A1} - y_{A2})}{z y_{B,lm}} \quad \text{---------- (1)}$$

where z equals $z_2 - z_1$, the length of the diffusion path at time t.

The molar flux N_A is related to the amount of A leaving the liquid by

$$N_A = \frac{\rho_{A,L}}{M_A} \frac{dz}{dt} \quad \text{---------- (2)}$$

where $\frac{\rho_{A,L}}{M_A}$ is the molar density of A in the liquid phase

under Psuedo steady state conditions, equations (1) & (2) can be equated to give

$$\frac{\rho_{A,L}}{M_A} \frac{dz}{dt} = \frac{C D_{AB} (y_{A1} - y_{A2})}{z y_{B,lm}} \quad \text{---------- (3)}$$

Equation. (3) may be integrated from t = 0 to t and from $z = z_{t0}$ to $z = z_t$ as:

$$\int_{t=0}^{t} dt = \frac{\rho_{A,L}\, y_{B,lm} / M_A}{C D_{AB} (y_{A1} - y_{A2})} \int_{Z_{t0}}^{Z_t} z\, dz$$

yielding

$$t = \frac{\rho_{A,L}\, y_{B,lm} / M_A}{C D_{AB} (y_{A1} - y_{A2})} \left(\frac{z_t^2 - z_{t0}^2}{2}\right) \quad \text{-------- (4)}$$

This shall be rearranged to evaluate diffusivity D_{AB} as,

$$D_{AB} = \frac{\rho_{A,L}\, y_{B,lm}}{M_A\, C\, (y_{A1} - y_{A2})\, t} \left(\frac{z_t^2 - z_{t0}^2}{2} \right)$$

Equimolar counter diffusion:

A physical situation which is encountered in the distillation of two constituents whose molar latent heats of vaporization are essentially equal, stipulates that the flux of one gaseous component is equal to but acting in the opposite direction from the other gaseous component; that is, $N_A = - N_B$.

The molar flux N_A, for a binary system at constant temperature and pressure is described by

$$N_A = -C D_{AB} \frac{dy_A}{dz} + y_A (N_A + N_B)$$

or
$$N_A = -D_{AB} \frac{dC_A}{dz} + y_A (N_A + N_B) \qquad \text{------- (1)}$$

with the substitution of $N_B = - N_A$, Equation (1) becomes,

$$N_A = -D_{AB} \frac{dC_A}{dz} \qquad \text{----------} \qquad (2)$$

For steady state diffusion Equation. (2) may be integrated, using the boundary conditions:

at $z = z_1$ $C_A = C_{A1}$
and $z = z_2$ $C_A = C_{A2}$

Giving,

$$N_A \int_{z_1}^{z_2} dz = -D_{AB} \int_{C_{A1}}^{C_{A2}} dC_A$$

from which

$$N_A = \frac{D_{AB}}{z_2 - z_1} (C_{A1} - C_{A2}) \qquad \text{---------- (3)}$$

For ideal gases, $C_A = \frac{n_A}{V} = \frac{p_A}{RT}$. Therefore Equation. (3) becomes

$$N_A = \frac{D_{AB}}{RT\,(z_2 - z_1)} (P_{A1} - P_{A2}) \qquad \text{---------- (4)}$$

This is the equation of molar flux for steady-state equimolar counter diffusion.

Concentration profile in this equimolar counter diffusion may be obtained from,

$$\frac{d}{dz}(N_A) = 0$$ (Since N_A is constant over the diffusion path).

And from equation. (2)

$$N_A = -D_{AB}\frac{dC_A}{dz}.$$

Therefore

$$\frac{d}{dz}\left(-D_{AB}\frac{dC_A}{dz}\right) = 0\ .$$

or $$\frac{d^2C_A}{dz^2} = 0.$$

This equation may be solved using the boundary conditions to give

$$\frac{C_A - C_{A1}}{C_{A1} - C_{A2}} = \frac{z - z_1}{z_1 - z_2}$$ ---------- (5)

Equation, (5) indicates a linear concentration profile for equimolar counter diffusion.

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UNIT – II SORPTION TECHNIQUES SCHA3005

SCHOOL OF BIO AND CHEMICAL ENGINEERING

DEPARTMENT OF CHEMICAL ENGINEERING

FILTRATION

Filtration plays an important role in the natural treatment of groundwater as it percolates through the soil. It is also a major part of most water treatment. Groundwater that has been softened, or treated through iron and manganese oxidation, requires filtration to remove floc created by coagulation or oxidation processes. Since surface water is subject to run-off and does not undergo natural filtration, it must be filtered to remove particles and impurities.

Filtration can be compared to a sieve or micro-strainer that traps suspended material between the grains of filter media. However, since most suspended particles can easily pass through the spaces between grains of the filter media, straining is the least important process in filtration. Filtration primarily depends on a combination of complex physical and chemical mechanisms, the most important being adsorption. Adsorption is the process of particles sticking onto the surface of the individual filter grains or onto the previously deposited materials. Forces that attract and hold particles to the grains are the same as those that work in coagulation and flocculation. In fact, coagulation and flocculation may occur in the filter bed, especially if coagulation and flocculation before filtration was not properly controlled. Incomplete coagulation can cause serious problems in filter operation.

Theory of filtration

Depending on dispersing medium filtration is divided in two parts: 1) gas filtration and 2) liquid filtration. Gas filtration theory It mainly includes filtration of aerosols and lyosols. Membrane filters and nucleopore filters are based on these below mechanisms. Mechanism of gas filtration (Wilson & Cavanagh,1969) Diffusion deposition: The trajectories of individual small particles do not coincide with the streamlines of the fluid

because of Brownian motion. With decreasing particle size the intensity of Brownian motion increases and, as a consequence, so does the intensity of diffusion deposition. Direct interception: This mechanism involves the finite size of particles. A particle is intercepted as it approaches the collecting surface to a distance equal to its radius. A special case of this mechanism is the so-called sieve effect, or sieve mechanism. Inertial deposition: The presence of a body in the flowing fluid results in a curvature of the streamlines in the neighborhood ofthe body. Because of their inertia, the individual particles do not follow the curved streamlines but are projected against the body and may deposit there. It is obvious that the intensity of this mechanism increases with increasing particlesize and velocity of flow. Gravitational deposition: Individual particles have a certain sedimentation velocity due to gravity. As a consequence, the particles deviate from the streamlines of the fluid and, owing to this deviation; the particles may touch a fiber. Electrostatic deposition: Both the particles and the fibers in the filter may carry electric charges. Deposition of particles on the fibersmay take place because of the forces acting between charges or induced forces. Liquid filtration theory (Melia & Weber, 1972). The term solid-liquid filtration covers all processes in which a liquid containing suspended solid is freed of someor the entire solid when the suspension is drawn through a porous medium.

Kozeny-Carman equation: $Idv/Adt= \Delta P/ r \mu (l+L)$

Where, A = filter area; V = total volume of filtrate delivered; t = filtration time; ΔP = pressure drop across cake and medium; r = specific cake resistance; μ = filtrate viscosity; l = cake thickness; L = thickness of cake equivalent to medium resistance.

TYPES OF FILTERS

Several types of filters are used for water treatment. Early slow sand filters typically have filter rates of 0.05 gpm/ft2 of surface area and require large filter areas. The top several inches of the sand has to be removed regularly-- usually by hand--due to the mass of growing material „schmutzdecke" that collects in the filter. Sand removed is usually washed and returned to the filter. These filters are still in use in some small plants, especially in the western United States as well as in many developing countries. They may also be used as a final step in wastewater treatment.Modern filters are classified as: Gravity Filters (Rapid Sand or High Rate-Dualmedia-Multi- media) or Pressure Filters (Sand or Multi-

media).

RAPID SAND FILTERS

Rapid sand filters have filter rates 40 times those of slow sand filters. The major parts of a rapid sand filter are:

•Filter tank or filter box

•Filter sand or mixed-media

•Gravel support bed

•Underdrain system

•Wash water troughs

•Filter bed agitators

The filter tank is generally constructed of concrete and is most often rectangular. Filters in large plants are usually constructed next to each other in a row, allowing piping from the sedimentation basins to feed the filters from the central pipe gallery. Some smaller plants are designed with filters forming a square of four filters with a central pipe gallery feeding the filters from a center wall.

Filter Sand

The filter sand used in rapid sand filters is manufactured specifically for the purpose of water filtration. Most rapid sand filters contain 24-30 inches of sand, but some newer filters are deeper. The sand used is generally 0.4 to 0.6 mm in diameter. This is larger than the sand used in slow rate filtration. The coarser sand in the rapid filters has larger voids that do not fill as easily.

PRESSURE FILTERS

Pressure filters fall into two categories: pressure sand and diatomaceous earth filters. Pressure filters are used extensively in iron and manganese removal plants. A pressure sand filter is contained under pressure in a steel tank, which may be vertical or horizontal, depending on the space available. As with gravity filters, the media is usually sand or a combination of media, and filtration ratesare similar to gravity filters.

Groundwater is first aerated to oxidize the iron or manganese, and then pumped through the filter to remove the suspended material.

FILTER AIDS

When water passes through a filter, the floc sometimes is torn apart into smaller particles that penetrate deeply into the filter media, causing premature turbidity breakthrough. This requires more frequent filter backwashing of the filter and large volumes of backwash water to be able to remove the floc that has penetrated deeply into the filter bed.

A filter aid is a material that adds strength to the floc and prevents its breakup. Generally, a polymer is used as a filter aid because it creates strong bonds with the floc. Polymers are water soluble, organic compounds that can be purchased in either wet or dry form.

Polymers have very high molecular weight and cause the floc to coagulate and flocculate quickly. Polymers can have positive or negative charges, depending on the type needed to cause attraction to the specific floc filtered.

When used as a filter aid, the polymer strengthens the bonds and prevents the shearing forces in the filter from breaking the floc apart. For best results, the polymer should be added just ahead of the filter. A normal dose of polymer for filter aiding will be less than 0.1 ppm, but the exact dose will be decided by the result of a jar test and by experimentation in the treatment plant. Too much polymer will cause the bonds to become too strong, which may then cause the filter to plug, especially the top few inches of the filter media.

COAGULATION AND FLOCCULATION PROCESS FUNDAMENTALS

Coagulation and Flocculation Groundwater and surface water contain both dissolved and suspended particles. Coagulation and flocculation are used to separate the suspended solids portion from the water. Suspended particles vary in source, charge, particle size, shape, and density. Correct application ofcoagulation and flocculation depends upon these factors. Suspended solids in water have a negative charge and since they have the same type of surface charge, they repel each other when they come close together. Therefore, suspended solids will remain in suspension and will not clump together and settleout of the water, unless proper coagulation and flocculation is used. Coagulation and flocculation occurs in successive steps, allowing particle collision and growth of floc. This is

then followed by sedimentation (see Sedimentation Chapter). If coagulation is incomplete, flocculation step will be unsuccessful, and if flocculation is incomplete, sedimentation will be unsuccessful.

COAGULATION Coagulant chemicals with charges opposite those of the suspended solids are added to the water to neutralize the negative charges on non-settlable solids (such as clay and color-producing organic substances). Once the charge is neutralized, the small suspended particles are capable of sticking together. These slightly larger particles are called microflocs, and are not visible to the naked eye. Water surrounding the newly formed microflocs should beclear. If not, coagulation and some of the particles charge have not been neutralized. More coagulant chemicals may need to be added. A high-energy, rapid-mix to properly disperse coagulant and promote particle collisions is needed to achieve good coagulation. Over-mixing does not affect coagulation,but insufficient mixing will leave this step incomplete. Contact time in the rapid- mix chamber is typically 1 to 3 minutes.

Alum $A1_2(SO_4)_3 + 3\ Ca(HCO_3)_2$ ---------- > $2\ Al(OH)_3 + 3CaSO_4 + 6\ CO_2$ Aluminum + Calcium gives Aluminum + Calcium + Carbon Sulfate Bicarbonate Hydroxide Sulfate Dioxide (present in the water to treat) Ferric Sulfate $Fe2(SO4)3 + 3\ Ca(HCO_3)_2$ ----------> $2\ Fe(OH)_3 + 3CaSO4 + 6\ CO_2$ Ferric + Calcium gives Ferric + Calcium + Carbon Sulfate Bicarbonate Hydroxide Sulfate Dioxide (present in the water to treat) Ferric Chloride $2\ Fe\ Cl_3 + 3\ Ca(HCO_3)_2$ ----------> $2\ Fe(OH)_3 + 3CaCl_2 + 6CO_2$ Ferric + Calcium gives Ferric + Calcium + Carbon Chloride Bicarbonate Hydroxide Chloride Dioxide (present in the water to treat) Polymers Polymers (long-chained, high-molecular-weight, organic chemicals) are becoming more widely used. These can be used as coagulant aids along with the regular inorganic coagulants. Anionic (negatively charged) polymers are often used with metal coagulants. Low-to-medium weight cationic (positively charged) polymers may be used alone, or in combination with alumor ferric coagulants to attract suspended solids and neutralize their surface charge. Manufacturers can produce a wide range of polymers that meet a variety of source-water conditions by controlling the amount and type of charge and the polymers molecular weight. Polymers are effective over a wider pH range than inorganic coagulants. They can be applied at lower doses, and do not consume alkalinity. They produce smaller volumes of more concentrated, rapidly settling

floc. Floc formed from use of a properly selected polymer will be more resistant to shear, resulting in less carryover and a cleaner effluent. Polymers are generally several times more expensive in price per pound than inorganic coagulants. Selection of the proper polymer requires considerable jar testing under simulated plant conditions, followed by pilot or plant-scale trials. All polymers must also be approved for potable water use by regulatory agencies.

Precipitation:

Precipitation is the most commonly used technique in industry for the concentration of macromolecules such as proteins and polysaccharides. Further, precipitation technique can also be employed for the removal of certain unwanted byproducts e.g. nucleic acids, pigments.

Neutral salts, organic solvents, high molecular weight polymers (ionic or non-ionic), besides alteration in temperature and pH are used in precipitation. In addition to these non-specific protein precipitation reactions (i.e. the nature of the protein is unimportant), there are some protein specific precipitations e.g., affinity precipitation, ligand precipitation.

Neutral salts:

The most commonly used salt is ammonium sulfate, since it is highly soluble, nontoxic to proteins and low-priced. Ammonium sulfate increases hydrophobic interactions between protein molecules that result in their precipitation. The precipitation of proteins is dependent on several factors such as protein concentration, pH and temperature.

Organic solvents:

Ethanol, acetone and propanol are the commonly used organic solvents for protein precipitation. They reduce the dielectric constant of the medium and enhance electrostatic interaction between protein molecules that lead to precipitation. Since proteins are denatured by organic solvents, the precipitation process has to be carried out below 0°C.

Non-ionic polymers:

Polyethylene glycol (PEG) is a high molecular weight non-ionic polymer that can precipitate proteins. It reduces the quantity of water available for protein solvation and precipitates protein. PEG does not denature proteins, besides being non-toxic.

Ionic polymers:

The charged polymers such as polyacrylic acid and polyethyleneimine are used. They form complexes with oppositely charged protein molecules that causes charge neutralisation and precepitation.

Increase in temperature:

The heat sensitive proteins can be precipitated by increasing the temperature.

Change in pH:

Alterations in pH can also lead to protein precipitation.

Affinity precipitation:

The affinity interaction (e.g., between antigen and antibody) is exploited for precipitation of proteins.

Precipitation by ligands:

Ligands with specific binding sites for proteins have been successfully used for selective precipitation.

Cell Disruption:

Physical methods of cell disruption:

The microorganisms or cells can be disrupted by certain physical methods to release the intracellular products.

Ultra sonication:

Ultrasonic disintegration is widely employed in the laboratory. However, due to high cost, it is not suitable for large-scale use in industries.

Osmotic shock:

This method involves the suspension of cells (free from growth medium) in 20% buffered sucrose. The cells are then transferred to water at about 4°C. Osmotic shock is used for the release of hydrolytic enzymes and binding proteins from Gram-negative bacteria.

Heat shock (thermolysis):

Breakage of cells by subjecting them to heat is relatively easy and cheap. But this technique can be used only for a very few heat-stable intracellular products.

High pressure homogenization:

This technique involves forcing of cell suspension at high pressure through a very narrow orifice to come out to atmospheric pressure. This sudden release of high pressure creates a liquid shear that can break the cells.

Impingement:

In this procedure, a stream of suspended cells at high velocity and pressure are forced to hit either a stationary surface or a second stream of suspended cells (impinge literally means to strike or hit). The cells are disrupted by the forces created at the point of contact. Micro fluidizer is a device developed based on the principle of impingement. It has been successfully used for breaking E. coli cells. The advantage with impingement technique is that it can be effectively used for disrupting cells even at a low concentration.

Grinding with glass beads:

The cells mixed with glass beads are subjected to a very high speed in a reaction vessel. The cells break as they are forced against the wall of the vessel by the beads. Several factors influence the cell breakage-size and quantity of the glass beads, concentration and age of cells, temperature and agitator speed. Under optimal conditions, one can expect a maximal breakage of about 80% of the cells. A diagrammatic representation of a cell disrupter employing glass beeds is shownin Fig. 20.6. It contains a cylindrical body with an inlet, outlet and a central motor-driven shaft. To this shaft are fitted radial agitators. The cylinder is fitted with glass beads. The cell suspension is added through the inlet and the disrupted cells come out through the outlet. The body of the cell disrupter is kept cool while the operation is on.

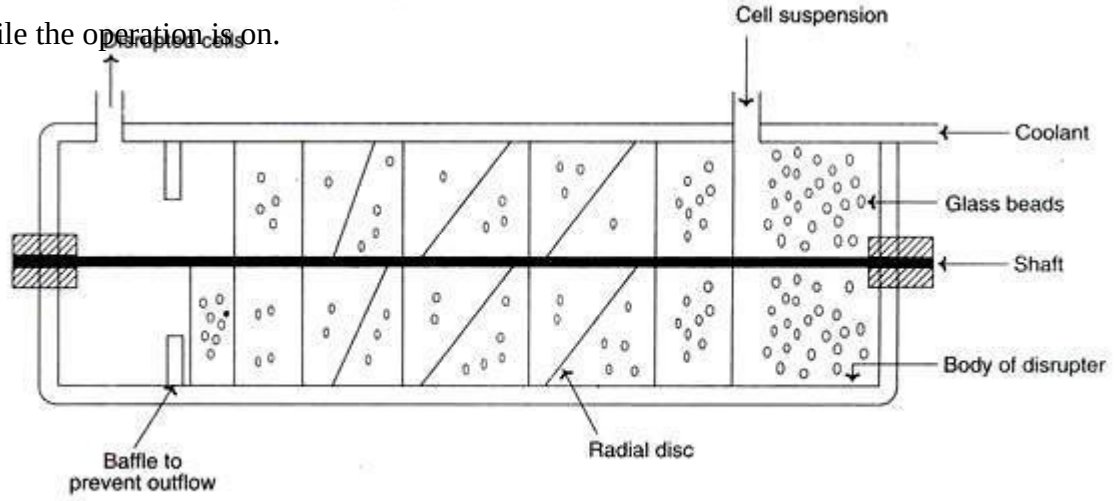


Fig. 20.6 : Diagrammatic representation of a cell disrupter.

Mechanical and non-mechanical methods:

Among the physical methods of cell disruption described above, ultra sonication, high-pressure homogenization, impingement and grinding with glass beads are mechanical while osmotic shock and heat shock are non-mechanical. The chemical and enzymatic methods (described below) are non- mechanical in nature.

Chemical methods of cell disruption:

Treatment with alkalies, organic solvents and detergents can lyse the cells to release the contents.

Alkalies:

Alkali treatment has been used for the extraction of some bacterial proteins. However, the alkali stability of the desired product is very crucial for the success of this method e.g., recombinant growth hormone can be efficiently released from E. coli by treatment with sodium hydroxide at pH 11.

Organic solvents:

Several water miscible organic solvents can be used to disrupt the cells e.g., methanol, ethanol, isopropanol, butanol. These compounds are inflammable; hence require specialised equipment for fire safety. The organic solvent toluene is frequently used. It is believed that toluene dissolves membrane phospholipids and creates membrane pores for release of intracellular contents.

Detergents:

Detergents that are ionic in nature, cationic-cetyl trimethyl ammonium bromide or anionic-sodium lauryl sulfate can denature membrane proteins and lyse the cells. Non-ionic detergents (although less reactive than ionic ones) are also used to some extent e.g., Triton X-100 or Tween. The problem with the use of detergents is that they affect purification steps, particularly the salt precipitation. This limitation can be overcome by using ultrafiltration or ion-exchange chromato- graphy for purification.

Enzymatic methods of cell disruption:

Cell disruption by enzymatic methods has certain advantages i.e., lysis of cells occurs under mild conditions in a selective manner. This is quite advantageous for product recovery. Lysozyme is the most frequently used enzyme and is commercially available (produced from hen egg white). It hydrolyses β-1, 4-glycosidic bonds of the mucopeptide in bacterial cell walls. The Gram- positive

bacteria (with high content of cell wall mucopeptides) are more susceptible for the action of lysozyme.

For Gram-negative bacteria, lysozyme in association with EDTA can break the cells. As the cell wall gets digested by lysozyme, the osmotic effects break the periplasmic membrane to release the intracellular contents. Certain other enzymes are also used, although less frequently, for cell disruption. For the lysis of yeast cell walls, glucanase and mannanase in combination with proteases are used.

AQUEOUS TWO-PHASE EXTRACTION

Aqueous two-phase extraction which is a special case of liquid-liquid extraction involves transfer of solute from one aqueous phase to another. The two immiscible aqueous phases are generated in-situ by addition of substances suchas polymers and salts to an aqueous solution. Historically, gelatin-agar and gelatin- soluble starch were used. Two types of aqueous two-phase systems are commonly used:

1.Polymer-polymer two-phase system 2.Polymer-salt two-phase system

Tab. 1. Two-Phase Aqueous Polymer Systems

Polymer/Salt Combination
Polyethylene glycol (PEG)-dextran
PEG-phosphate
PEG-citrate
PEG-hydroxypropyl starch
Ethylhydroxyethyl cellulose-hydroxypropyl starch
PEG-polyvinyl alcohol
PEG-pullulan
PEG-maltodextrins

A polymer-polymer two phase system can for instance be obtained by mixing dextran and PEG at a certain composition. By adding specific amounts of these polymers to an aqueous feed phase (which contains the solute), two aqueous phases, one rich in PEG and the other rich in dextran can be obtained. Aqueous two-phase systems can also be generated using a polymer (e.g. PEG or

dextran) and a salt such as sodium or potassium phosphate. Aqueous two- phase separations take place at certain compositions only. The figure belowshows a PEG-dextran phase diagram where a solubility curve separates the two- phase region (above the curve) from the single phase region (below the curve). Such "binary" phase diagrams which are based on the compositions of thetwo polymers (or polymer and salt) are used for determining the concentrations needed for an extraction process. These phase diagrams also predict the polymer/salt content of the raffinate and the extract phases. The composition of the individual phases generated can be obtained using tie-lines as shown in the

Biomolecule	Recovery (%)
Xylanase	41
β-glucosidase	85 - 95
Polyphenol oxidase	50
Lipase	68
β-galactosidase	90
Glucoamylase	96
Horseradish peroxidase	75
Thaumatin	90 - 95
β-galactosidase	85 - 100
Fumarate hydratase (*Brevibacterium ammoniagenes*)	83
Fumarate hydratase (*E. coli*)	93
Aspartate-ammonia-lyase (*E. coli*)	96

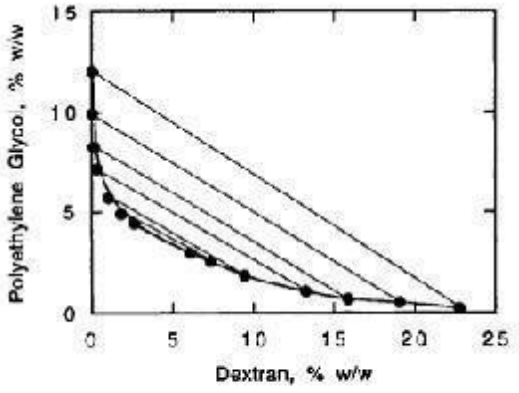


Fig. 2. Phase diagram for a PEG 6000-dextran D48 two-phase system at 20 °C (redrawn from Albertsson, 1986).

figure.

The partition of a solute between the two aqueous phases depends on its physicochemical properties as well as those of the two polymers (or polymer and salt).Factors Affecting Protein Partitioning in Two-Phase Aqueous Polymer Systems

1.Protein molecular weight

2.Protein charge, surfaceproperties

3.Polymer molecular weight

4.Phase composition

5. Salt effects

6. Affinity ligands attached to polymers 7.pH

8.Temperature

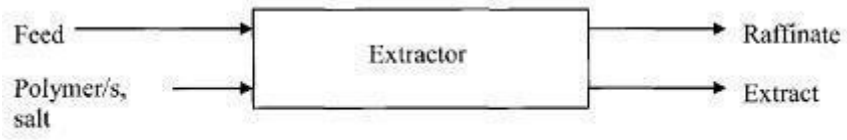


Fig. 7.4 Aqueous two-phase extraction

In PEG/dextran aqueous two-phase extraction of proteins, the partition behavior depends to a great extent on the relative polymer composition. It also depends on the solution pH and the molecular weight of the protein. Generally speaking, protein partitioning into the PEG rich phase is favored. When a polymer- salt combination is used to generate the aqueous two-phase system, a protein partitions favorably into the polymer rich phase. The general scheme for aqueous two-phase extraction is shown in Fig. 7.4.Extraction by an ATPS offers advantages for processing on a large scale, such as the possibility of obtaining a high yield, the possibility of continuous processing and a reduction in operationalcost in relation to the costs of conventional processes.

PEG-DEXTRAN SYSTEMS

Effect of Polymer Molecular Mass (MM)

An increase in the molecular mass of dextran or of PEG will lower the concentration required for phase separation. The polymer molecular mass influences protein partitioning as a direct result of interactions between the two polymers. It has been found that the partitioned protein behaves as if it were more attracted by smaller polymer sizes and more repelled by larger polymers,

provided all other factors such as polymer concentrations, salt composition, temperature and pH are kept constant. It was observed that smaller protein molecules and amino acids were not affected as much as larger ones. For some proteins the partition coefficients increased as the MM of dextran increased if all other conditions were kept constant, but little effect was found for low MM proteins (Cytochrome C, 16,000). When the same proteins were partitioned in systems with different PEG MM, their partition coefficients decreased as the PEG MM increased, and for cytochrome C the effect was the smallest. This was attributed to the fact that when the PEG MM is increased, a weaker repulsion energy is required to cause phase separation. Repulsive interactions between the polymer and the protein become stronger as the polymer MM is increased,resulting in a distribution of the protein towards the phase containing the polymerwith an unchanged MM. A weak net repulsion between the proteins and the polymer is sufficient to change the distribution when the polymer MM is changed.

Effect of Polymer Concentration

It was found that proteins with MM less than 20,000 showed a linear relationship between the ln K in PEG-dextran systems and a difference in PEG concentration between the phases, for any particular system. They found that it was possible to predict the partitioning of a protein at any concentration in that particular system if one partition coefficient in the system were known.

However, others found that for some proteins the partition coefficient was inversely correlated to phase concentration in a PEG-dextran system, showing that better separation could be achieved at high polymer concentrations. This, however, may also affect the concentration of proteins that can be manipulated in the system as polymer concentration has a directly inverse effect on protein solubility.

Effect of Salts

Salts can affect protein partitioning in different ways in PEG-dextran systems: one is by altering the physical properties of the systems the hydrophobic difference between the phases and the other is by the partitioning of ions betweenthe phases, which affects the partitioning of proteins according to their molecular charge.

Salts have been added to PEG-dextran systems to increase the selectivity of

protein partitioning in the aqueous two-phase methodology application for biological separations. It was observed that salt ions partition differently between the phases, causing an uneven distribution in the system that generates a difference in electrical potential between the phases. This difference in electrical potential would be independent of salt concentration, but linearly dependent on the partition behaviour of the ions. It was also observed that polyvalent anions such as phosphate, sulphate and citrate partitioned preferentially into dextran-richphases, while halides partitioned nearly equally. As an example, negatively charged materials have higher partition coefficients in phases containing sodium sulphate rather than sodium chloride, while the reverse holds for positively charged materials. Partition coefficients of negatively charged materials decrease when the cationic series is changed from lithium to sodium to potassium. The ratio between the phosphate ions, rather than the concentration, was decisive for the difference in electrical potential. This applies to multivalent ions, which showa series of pH-dependent dissociations and was clearly the reason for the potential difference found between the two phases (Kula et al., 1982).

PEG-SALT SYSTEMS

The formation of PEG-salt systems was first observed in the 1950s, but the theoretical fundamentals have not been well explained. It was found that for PEG solutions the addition of some inorganic salts (sulphates and carbonates) is more effective than the addition of others in reducing the critical concentration of cloud point curves. These inorganic salts dramatically reduced the PEG cloud point at high temperatures.

PEG-salts systems have been introduced for the practical application of large-scaleprotein separation because of the larger droplet size, greater difference in density between the phases, lower viscosity and lower costs, leading to a much faster separation than in PEG-dextran systems. Industrial application of PEG-

Biomolecule	Ligands attached to PEG	Recovery (%)
Lactate dehydrogenase	Tryazine dye-Cibacron Blue F3G-A	81.3
β-galactosidase	p-amino phenyl-β-D-thiogalactopiranoside – (APGP)	83
Protein A	IgG human	87
Lactate dehydrogenase	Eudrogit-Cibacron Blue	54
Penicillin acylase	Trimethylamina	97
Trypsin	Trypsin inhibitor	82

was improved by the availability of commercial separators, which allowed faster continuous protein.

Initially PEG-phosphate systems were widely used where scientists have studied ways of recycling the phosphate phase of the systems to minimize environmental pollution. The recycling of the phosphate phase was achieved by its separation from the solids by the use of alcohols. PEG from the top PEG-rich phase can also be successfully recycled.

More recently PEG-sulphate systems have begun to be used where separation of some biomaterial was achieved with PEG 4000 and (NH4)2SO4 at pH 7-7.5. The presence of 2% NaCl (0.17 M) made the separation much worse. With 4% NaCl (0.34 M), a poor separation was obtained (a tenfold decrease in K for aspartase). Since a pH or phase ratio change was not observed, the dramatic change in K was considered to be due to a change in hydrophobicity between the phases.

AFFINITY PARTITIONING

In the last 30 years, several groups have studied methods to increase partitioning by the use of biospecific interactions in ATPSs.

The initial works on affinity partitioning in ATPSs were to purify trypsin by usingPEG-bound ligand p-aminobenzamidine and S-23 myeloma protein by using dinitrophenol as ligand.The degree of affinity partitioning, Kaff, can be described by the ratio between the partition coefficients of proteins with and without a ligand:

This equation describes the increase in the partition coefficient of a protein by the binding of a specific ligand to the PEG-rich phase.

Affinity partitioning results in specific extractions of proteins, nucleic acids, membranes, organelles and even cells, mainly when biospecific ligands are used.

Large Scale Extraction Schemes

Extraction processes can be divided into two general schemes:

•Batch extractions

•Continuous extractions,

Continuous extractions can also be further divided into the following schemes:

•Single stage continuous extraction

•Multi stage continuous extraction

In turn, multi stage continuous extraction can be divided into two general modes as:

•Crosscurrent continuous extraction

•Counter current continuous extraction These will be studied in the following sections.

Batch extraction

In a batch extraction process a batch of feed solution is mixed with a batch of extracting solvent in an appropriate vessel. The solute distributes between the two phases depending on its partition coefficient. The rate at which the transfer of solute takes place from the feed to the extracting solvent depends on the mixing rate. Once equilibrium is attained, the mixing is stopped and the extract and raffinate phases are allowed to separate. The separation funnel commonly seen in chemistry laboratories is the simplest small-scale batch extraction device. Mixer-settler units are usually used for large-scale batch extraction. The basic principle of batch extraction using a mixer settler unit is shown in Fig. 7.5. The mixer unit must be able to generate high interfacial area, must provide high solute mass transfer coefficient and cause low entrainment of air bubbles. The settler unit must have a low aspect ratio (L/D), i.e. be of flat geometry, must allow easy coalescence and phase separation, and must allow for easy collection of raffinate and extract as separate streams. The antibiotic penicillin partitions favorably in an

organic solvent from an aqueous fermentation media at acidic conditions. However, at a neutral pH, the partitioning from organic phase to aqueous phase is favored. Thus the antibiotic could be purified by sequential reversed batch extraction, where the antibiotic is moved from aqueous to organic phase and back again (as shown in Fig. 7.6). This sequence is usually repeated a few times in order to obtain highly pure antibiotic.

If a batch of feed containing R volume of initial solvent and an initial solute concentration of C0 is mixed with S volume of pure extracting solvent, the concentration distribution in the extract and the raffinate at equilibrium is given by:

CE = KCR

Where

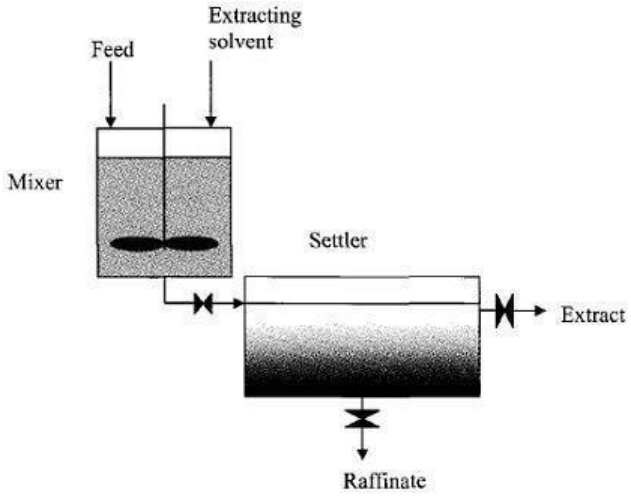


Fig. 7.5 Batch extraction using mixer settler unit

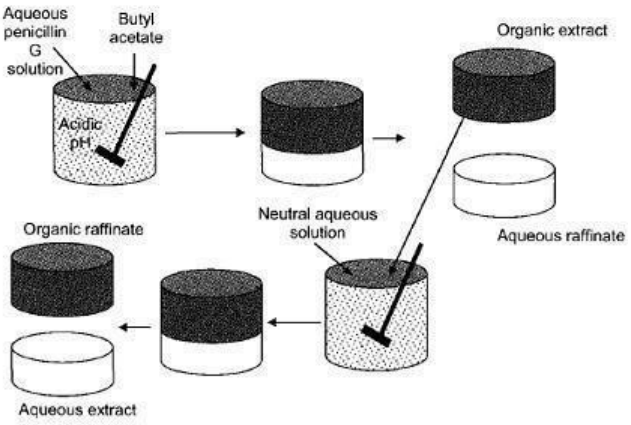


Fig. 7.6 Sequential reverse batch extraction

CE solute concentration in extract (kg/m3) CR solute concentration in raffinate (kg/m3)

Supercritical Fluid Extraction (SFE) is the process of separating one component (the extractant) from another (the matrix) using supercritical fluids as the extracting solvent. Extraction is usually from a solid matrix, but can also be from liquids. SFE can be used as a sample preparation step for analytical purposes, or on a larger scale to either strip unwanted material froma product (e.g. decaffeination) or collect a desired product (e.g. essential oils). These essential oils can include limonene and other straight solvents. Carbon dioxide (CO_2) is the most used supercritical fluid, sometimes modified by co- solvents such as ethanol or methanol. Extraction conditions for Supercritical carbon dioxide are above the critical temperature of 31°C and critical pressure of 74 bar. Addition of modifiers may slightly alter this. The discussion below will mainly refer to extraction with CO_2, except where specified. The properties of a supercritical fluid can be altered by varying the pressure and temperature, allowing selective extraction. For example, volatile oils can be extracted from a plant with low pressures (100 bar), whereas liquid extraction would also remove lipids. Lipids can be removed using pure CO_2 at higher pressures, and then phospholipids can be removed by adding ethanol to the solvent. The same principle can be used to extract polyphenold and unsaturated fatty acids separately from wine wastes.

The system must contain a pump for the CO_2, a pressure cell to contain the sample, a means of maintaining pressure in the system and a collecting vessel. The liquid is pumped to a heating zone, where it is heated to supercritical conditions. It then passes into the extraction vessel, where it rapidly diffuses into the solid matrix and dissolves the material to be extracted. The dissolved materialis swept from the extraction cell into a separator at lower pressure, and the extracted material settles out. The CO_2 can then be cooled, re-compressed and recycled, or discharged to atmosphere.

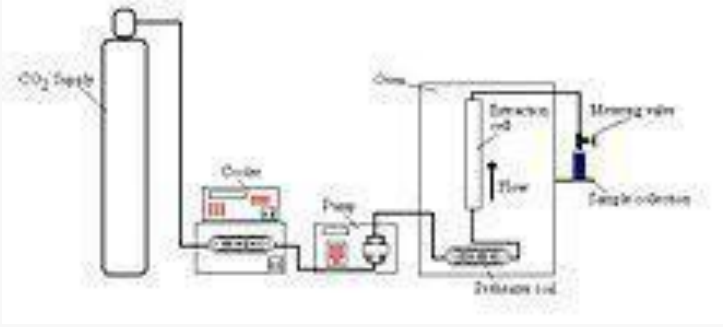


Figure 1. Schematic diagram of SFE apparatus

What Is adsorption chromatography?

Adsorption chromatography is the oldest types of chromatography technique. It makes use of a mobile phase which is either in liquid or gaseous form. The mobile phase is adsorbed onto the surface of a stationary solid phase.

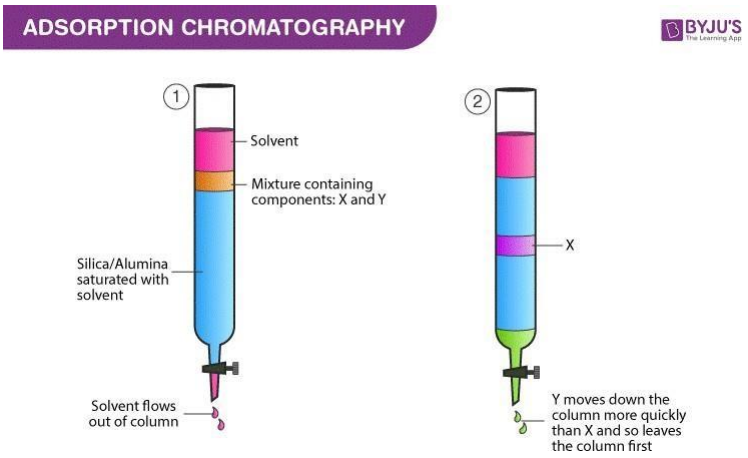
Adsorption Chromatography Principle

Adsorption Chromatography involves the analytical separation of a chemical mixture based on the interaction of the adsorbate with the adsorbent. The mixture of gas or liquid gets separated when it passes over the adsorbent bed that adsorbs different compounds at different rates.

Adsorbent – A substance which is generally porous in nature with a high surface area to adsorb substances on its surface by intermolecular forces is called

adsorbent. Some commonly used adsorbents are Silica gel H, silica gel G, silica gel N, silica gel S, hydrated gel silica, cellulose microcrystalline, alumina, modified silica gel, etc.

Adsorption Chromatography Diagram



Adsorption Chromatography Procedure

Before starting with the adsorption chromatography Experiment let us understand the two types of phases and the types of forces involved during the mixture separation process.

- Stationary phase – Adsorbent is the stationary phase in adsorption chromatography. The forces involved help to remove solutes from the adsorbent so that they can move with the mobile phase.

- Mobile phase – Either a liquid or a gas is used as a mobile phase in adsorption chromatography. Forces involved help to remove solutes from the adsorbent so that they can move with the mobile phase. When a liquid is used as a mobile phase it is called LSC (Liquid-Solid Chromatography). When a gas is used as a mobile phase it is called GSC (Gas-Solid Chromatography).

  Apparatus –

  Chromatography jar – The glass jar has a lid. It helps to maintain a proper environment during separation.

  Thin layer chromatography plate – Borosilicate glass plate with size 20*20 cm, 20*5 cm, 20*10.

  Capillary tube – Sample mixture is applied to TLC with the help of this tube.
  Mobile phase – Liquid or gas

  Stationary phase – Adsorbents

  Adsorption Chromatography Experiment (TLC)

- Take a clean and dry chromatographic jar.
- To make sure that the environment in the jar is saturated with solvent vapors, a paper soaked in the mobile phase is applied to the walls.
- Add the mobile phase to the jar and close it.
- Maintain equilibrium
- Mark the baseline on the adsorbent.
- Apply sample to TLC plate with the help of a capillary tube and allow it to dry.
- Put the plates in the jar and close it.
- Wait until the solvent moves from the baseline.
- Take out the TLC plate and dry it.

  Adsorption Chromatography

  Applications

- Adsorption chromatography is used for separation of amino acids.
- It is used in the isolation of antibiotics.
- It is used in the identification of carbohydrates.
- It is used to separate and identify fats and fatty acids.
- It is used to isolate and determine the peptides and proteins. Types of Adsorption Chromatography:

1. Thin Layer Chromatography – It is a chromatography technique where the mobile phase moves over an adsorbent. The adsorbent is a thin layer which is applied to a

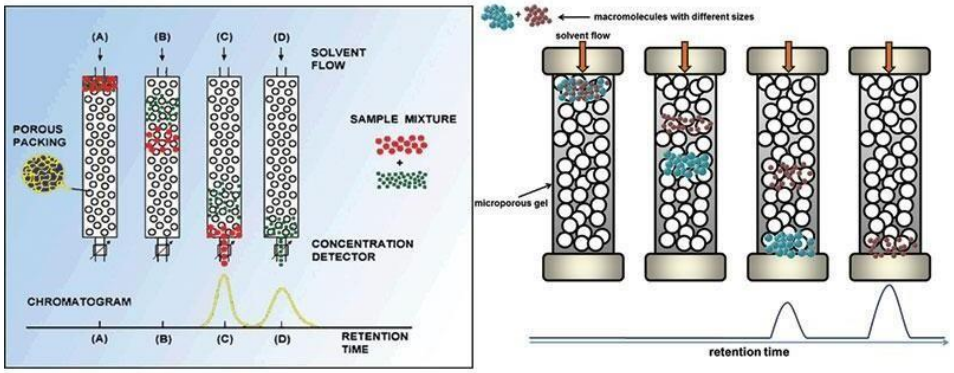
solid support for the separation of components. The separation takes place through differential migration which occurs when the solvent moves along the powder spread on the glass plates.

2. Mobile phase – This phase in TLC can either be a single liquid or mixture of liquids. Some commonly used liquids are Ethanol, acetone, methanol, chloroform. Stationary phase – Adsorbents
3. Column chromatography – the technique in which the solutes of a solution are entitled to travel down a column where the individual components are adsorbed by the stationary phase. Based on the affinity towards adsorbent the components take positions on the column. The most strongly adsorbed component is seen at the top of the column.
4. Gas-Solid chromatography – The principle of separation in GSC is adsorption. It is used for solutes which have less solubility in the stationary phase. This type of chromatography technique has a very limited number of stationary phases available and therefore GSC is not used widely.

Gel Permeation Chromatography

- Gel permeation chromatography is also called as gel filtration or size exclusion chromatography.
- In size exclusion chromatography, the stationary phase is a porous matrix made up of compounds like cross-linked polystyrene, cross-like dextrans, polyacrylamide gels, agarose gels, etc.
- The separation is based on the analyte molecular sizes since the gel behaves like a molecular sieve.
- This technique is used for the separation of proteins, polysaccharides, enzymes, and synthetic polymers.
- As a technique, size exclusion chromatography was first developed in 1955 by Lathe and Ruthven.

Gel Permeation Chromatography



Principle of Gel Permeation Chromatography

- It is a technique in which the separation of components is based on the difference in molecular weight or size.
- The stationary phase used is a porous polymer matrix whose pores are completely filled with the solvent to be used as the mobile phase.
- The molecules in the sample are pumped through specialized columns containing such microporous packing material (gel).
- The basis of the separation is that molecules above a certain size are totally excluded from the pores, while smaller molecules access the interior of the pores partly or wholly.
- The flow of the mobile phase hence will cause larger molecules to pass through the column unhindered, without penetrating the gel matrix, whereas smaller molecules will be retarded according to their penetration of the gel.

Components/ Instrumentation of Gel Permeation Chromatography

1. Stationary Phase
2. The Mobile Phase
3. The Columns
4. The Pump
5. Detectors

Stationary phase

It is composed of semi-permeable, porous polymer gel beads with a well-defined range of pore sizes.

It has the following properties:

- Chemically inert
- Mechanically stable
- With ideal and homogeneous porous structure (wide pore size give low resolution).
- A uniform particle and pore size. Examples of gel:

1. **Dextran** (Sephadex) gel: An α 1-6-polymer of glucose natural gel
2. **Agarose** gel: A 1,3 linked β-D-galactose and 1,4 linked 3,6-anhydro-α, L- galactose natural gel
3. **Acrylamide** gel: A polymerized acrylamide, a synthetic gel

B. The Mobile Phase

It is composed of a liquid used to dissolve the bio-molecules to make themobile phase permitting high detection response and wet the packing surface.

C. Columns

Any of the following kinds may be used:

- Analytical column- 7.5–8mm diameters.
- Preparative columns-22–25mm
- Usual column lengths-25, 30, 50, and 60 cm.
- Narrow-bore columns- 2–3mm diameter have been introduced

D. Pumps

They are either syringe pumps or reciprocating pumps with a high constant flow rate.

E. Detectors

The detectors may be concentration sensitive detectors, bulk property detectors, refractive index (RI) detector, etc.

Steps in Gel Permeation Chromatography

It involves three major steps:

A. Preparation of column for gel filtration

It involves:

1. Swelling of the gel
2. Packing the column semi-permeable, porous polymer gel beads with a well-defined range of pore sizes.
3. Washing: After packing, several column volumes of buffer solution is passed through the column to remove any air bubbles and to test the column homogeneity.

B. Loading the sample onto the column using a syringe

C. Eluting the sample and detection of components

Applications of Gel Permeation Chromatography

1. Proteins fractionation
2. Purification
3. Molecular weight determination.
4. Separation of sugar, proteins, peptides, rubbers, and others on the basis of their size.
5. Can be used to determine the quaternary structure of purified proteins

Advantages of Gel Permeation Chromatography

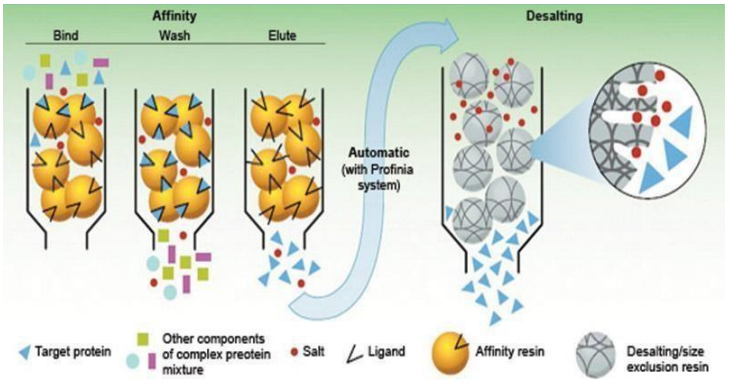
- Short analysis time.
- Well defined separation.
- Narrow bands and good sensitivity.
- There is no sample loss.
- The small amount of mobile phase required.
- The flow rate can be set

Limitations of Gel Permeation Chromatography

- The limited number of peaks that can be resolved within the short time scale of
- the GPC run.
- Filtrations must be performed before using the instrument to prevent dust and other particulates from ruining the columns and interfering with the detectors.
- The molecular masses of most of the chains will be too close for the GPC separation to show anything more than broad peaks.

Affinity Chromatography

- Chromatography is an important biophysical technique that enables the separation, identification, and purification of the components of a mixture for qualitative and quantitative analysis.
- It is a separation technique in which a mobile phase carrying a mixture is caused to move in contact with a selectively absorbent stationary phase.
- Affinity chromatography is a type of liquid **chromatography** for the separation, purification or specific analysis of sample components.
- It utilizes the reversible biological interaction or molecular recognition called affinity which refers to the attracting forced exerted in different degrees between atoms which cause them to remain in combination.
    - Example: Enzyme with and inhibitor, antigen with an antibody etc.
- It was discovered by Pedro Cuatrecasas and Meir Wilcheck.



- The stationary phase consists of a support medium, on which the substrate (ligand) is bound covalently, in such a way that the reactive groups that are essential for binding of the target molecule are exposed.
- As the crude mixture of the substances is passed through the chromatography

column, substances with binding site for the immobilized substrate bind to the stationary phase, while all other substances is eluted in the void volume of the column.

- Once the other substances are eluted, the bound target molecules can be eluted by methods such as including a competing ligand in the mobile phase or changing the pH, ionic strength or polarity conditions.

1. **Matrix**

- The matrix is an inert support to which a ligand can be directly or indirectly coupled.
- In order to for the matrix to be effective it must have certain characters:
- Matrix should be chemically and physically inert.
- It must be insoluble in solvents and buffers employed in the process
- It must be chemically and mechanically stable.
- It must be easily coupled to a ligand or spacer arm onto which the ligand can be attached.
- It must exhibit good flow properties and have a relatively large surface area for attachment.
- The most useful matrix materials are agarose and polyacrylamide.

2. **Spacer arm**

- It is used to improve binding between ligand and target molecule by overcoming any effects of steric hindrance.

3. **Ligand**

- It refers to the molecule that binds reversibly to a specific target molecule.
- The ligand can be selected only after the nature of the macromolecule tobe isolated is known.
- When a hormone receptor protein is to be purified by affinity chromatography, the hormone itself is an ideal candidate for the ligand.
- For antibody isolation, an antigen or hapten may be used as ligand.
- If an enzyme is to be purified,a substrate analog, inhibitor, cofactor, or effector may be used as a the immobilized ligand.

- Affinity medium is equilibrated in binding buffer.
- Sample is applied under conditions that favor specific binding of the target molecule(s) to a complementary binding substance (the ligand). Target substances bind specifically, but reversibly, to the ligand and unbound material washes

through the column.

- Elution is performed specifically, using a competitive ligand, or non-specifically, by changing the pH, ionic strength or polarity. Target protein is collected in a purified, concentrated form.
- Affinity medium is re-equilibrated with binding buffer.

These events can be summarized into the following three major steps:

1. **Preparation of Column**

- The column is loaded with solid support such as sepharose, agarose, cellulose etc.
- Ligand is selected according to the desired isolate.
- Spacer arm is attached between the ligand and solid support.

2. **Loading of Sample**

- Solution containing a mixture of substances is poured into the elution column and allowed to run at a controlled rate.

3. **Elution of Ligand-Molecule Complex**

- Target substance is recovered by changing conditions to favor elution of the bound molecules.

Applications of Affinity Chromatography

- Affinity chromatography is one of the most useful methods for the separation and purification of specific products.
- It is essentially a sample purification technique, used primarily for biological molecules such as proteins.

Its major application includes:

- Separation of mixture of compounds.
- Removal of impurities or in purification process.
- In enzyme assays
- Detection of substrates
- Investigation of binding sites of enzymes
- In in vitro antigen-antibody reactions
- Detection of Single Nuceotide polymorphisms and mutations in nucleic acids

Advantages of Affinity Chromatography

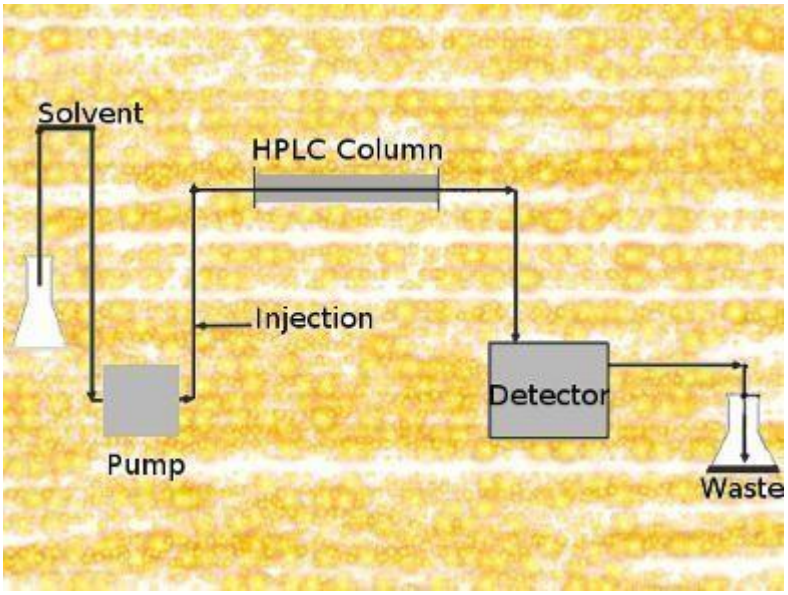
- High specificity
- Target molecules can be obtained in a highly pure state
- Single step purification

- The matrix can be reused rapidly.
- The matrix is a solid, can be easily washed and dried.
- Give purified product with high yield.
- Affinity chromatography can also be used to remove specific contaminants, such as proteases.

Limitations of Affinity Chromatography

- Time consuming method.
- More amounts of solvents are required which may be expensive.
- Intense labour
- Non-specific adsorption cannot be totally eliminated, it can only be minimized.
- Limited availability and high cost of immobilized ligands.
- Proteins get denatured if required pH is not adjusted.

HPLC



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The components of a mixture are separated from each other due to their different degrees of interaction with the absorbent particles. This causes different elution rates for the different components and leads to the separation of the components as they flow out the column. Compared to column chromatography, HPLC is highly automated and extremely sensitive. **Types of HPLC**

The two most common variants are normal-phase and reversed-phase HPLC.

Normal-Phase HPLC

The column is filled with tiny silica particles, and a non-polar solvent, for example,

hexane. A typical column has an internal diameter of 4.6 mm or smaller and a length of 150 to 250 mm. Non-polar compounds in the mixture will pass more quickly through the column, as polar compounds will stick longer to the polar silica than non-polar compound will.

Reversed-Phase HPLC

The column size is the same. The column is filled with silica particles which are modified to make them non-polar. This is done by attaching long hydrocarbon chains (8–18 C atoms) to its surface. A polar solvent is used, for example, a mixture of water and an alcohol such as methanol. Polar compounds in the mixture will pass more quickly through the column because a strong attraction occurs between the polar solvent and the polar molecules in the mixture.

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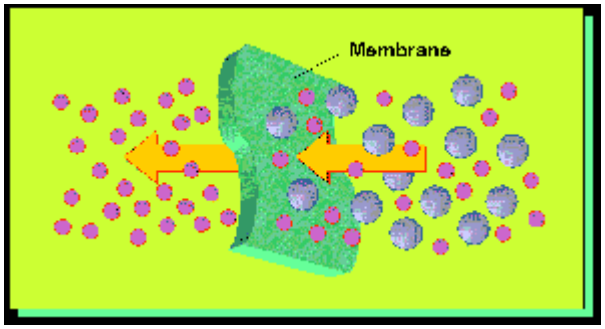
DEPARTMENT OF CHEMICAL ENGINEERING

UNIT – III- MEMBRANE SEPERATION SCHA3005

MEMBRANE SEPERATIONS

A membrane is a selective barrier that permits the separation of certain species in a fluid by combination of sieving and sorption diffusion mechanism. Separation is achieved by selectively passing (permeating) one or more components of a stream through the membrane while retarding the passage of one or more other components. See the Figure. Membranes can selectively separate components over a wide range of particle sizes and molecular weights, from macromolecular materials such as starch and protein to monovalent ions. Membranes have gained an important place in chemical technology and are used in a broad range of applications.

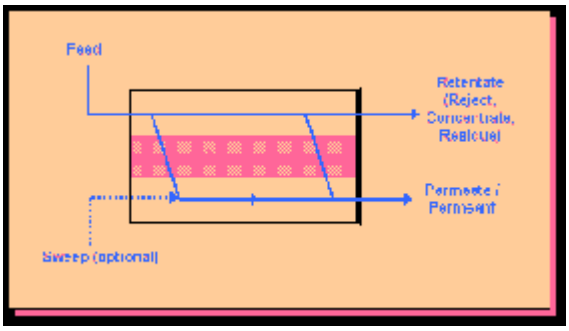


The key properties determining membrane performance are high selectivity and fluxes, good mechanical, chemical and thermal stability under operating conditions, low fouling tendencies and good compatibility with the operating environment; and cost effective and defect-free production.

Although the major uses of membranes are in the production of potable water and separation of industrial gases, they can be used for many other important applications such as filtration of particulate matter from liquid suspensions, air or industrial flue gas and the dehydration of ethanol azeotropes.

More specialised applications include ion separation in electrochemical processes, dialysis of blood and urine, artificial lungs, controlled release of therapeutic drugs, membrane-based sensors, etc.

Membrane processes are characterized by the fact that a feed stream is divided into 2 streams: retentate and permeate. The most general process can be depicted by the following Figure:



The retentate is that part of the feed that does not pass through the membrane, while the permeate is that part of the feed that does pass through the membrane. The optional "sweep" is a gas or liquid that is used to help remove thepermeate. The component(s) of interest in membrane separation is known as the solute. The solute can be retained on the membrane membrane and removed in the retentate or passed through the membrane in the permeate.

It is important to note that there are 3 different mechanisms by which membrane can perform separations:

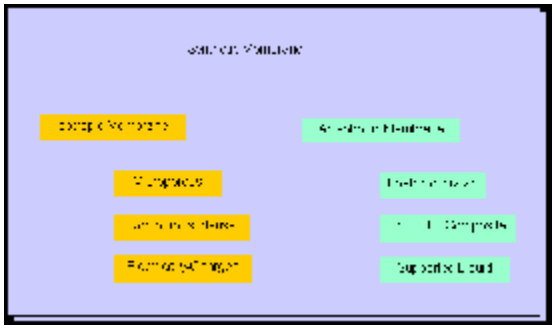
- CLASSIFICATION OF SYNTHETIC MEMBRANES

A membrane can be natural or synthetic, thick or thin, its structure can be homogeneous or heterogeneous, transport across membrane can be active or passive, passive transport can be driven by various means (e.g. pressure, concentration, electrical difference), neutral or charged. As such, membranes can

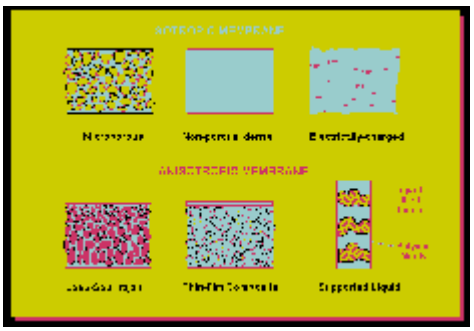
be classified according to different viewpoints. The first classification isby nature, i.e. biological or synthetic membranes. This is the clearest distinction possible. Synthetic membranes can be subdivided into organic (polymeric or liquid) and inorganic (e.g. ceramic, metal) membranes.

Another means of classifying membranes is by morphology or structure - for the case of solid synthetic membranes, the 2 types of membrane structures are the symmetric and asymmetric (anisotropic) membranes.

The principal types of polymeric membranes are classified as shown in the Figure below:



The Figure shows schematically the different types of membranes. This classification is, however, rather arbitrary, and many structures fit more than one of the above-mentioned classes. For example, a membrane may be microporous, asymmetric in structure, and have electrical charges.



The IUPAC classification for pores sizes are as follows:

Macropores	> 50 nm
Mesopores	2 – 50 nm
Micropores	< 2 nm

The following membranes are briefly discussed:

Isotropic Membranes

Microporous Membranes

Non-Porous, Dense Membranes

Electrically-Charged

Membranes

Anisotropic (Asymmetric) Membranes

Loeb-Sourirajan Membranes

Thin-Film (Composite)

Membranes Liquid Membranes

Membrane Materials

Almost all industrial membrane processes are made from natural or synthetic polymers. These membranes are sometimes known as organic membranes. Natural polymers include wool, rubber (polyisoprene) and cellulose. Examples ofsynthetic polymers include polyamide, polystyrene and polytetrafluoroethylene (Teflon).

Membranes can also be made from other non-polymeric materials. Such membranes include inorganic membranes (for example metal, ceramic, carbon and zeolites) and liquid membranes.

In addition, recent developments had led to the introduction of the so- called Hybrid Membranes (or Mixed Matrix Membranes, where both organic and inorganic components are used).

Another variation in membranes application is the Bipolar Membranes (BPM).

Polymeric Membranes

Synthetic polymers are produced by polymerisation of a monomer (condensation or addition) or by the co-polymerisation of 2 different monomers. The resulting polymer is categorised as having:

(1) a long, linear chain, such as linear polyethylene

(2) a branched chain, such as polybutadiene

(3) a three-dimensional highly cross-linked structure, such as phenol- formaldehyde

(4) moderately cross-linked structure, such as butyl rubber

The linear-chain polymers soften with an increase in temperature and are often soluble in organic solvents, and are referred to as thermoplastic polymers.

At the other extreme, high cross-linked polymers do not soften appreciably, are almost insoluble in most organic solvents, and are referred to as thermosetting polymers.

Material	Application					
	MF	UF	NF/RO	GS	PV	MD
Cellulose acetate	×	×	×	×	×	
Cellulose esters	×					
Cellulose nitrate	×					
Poly (vinyl alcohol)	×					
Polyacrylonitrile		×			×	
Poly (vinyl chloride)	×					
PVC copolymer	×	×				
Aromatic polyamide	×	×	×			
Aliphatic polyamide	×	×				
Polyimide	×	×	×	×		
Polysulfone	×	×				
Polyetheretherketone (PEEK)	×	×		×		
Polycarbonate	×					
Polyester	×					
Polypropylene	×				×	×
Polyethylene	×				×	×
Polytetrafluoroethylene (PTFE)	×	×			×	
Poly (vinylidene difluoride) (PVDF)	×	×			×	×
Polydimethylsiloxane (PDMS)				×	×	

Examples of polymers used in membrane fabrication are shown in the Figures below.

Inorganic Membranes

Inorganic membranes refer to membranes made of materials such as **ceramic, carbon, silica, zeolite, various oxides** (alumina, titania, zirconia) and **metals** such as palladium, silver and their alloys. Inorganic membranes can be classified into 2 major categories based on its structure: porous inorganic membranes and dense (non-porous) inorganic membranes. Microporous inorganic membranes have 2 different structures: symmetric and asymmetric; andinclude both amorphous and crystalline membranes.

Microporous inorganic membranes can be obtained by coating of a porous support with a colloidal solution, called **sol**. The sol can consist of either dense spherical

particles (colloids of oxides such as Al_2O_3, SiO_2 or ZrO_2) or polymeric macromolecules.

Application of dense inorganic membranes is primarily for highly selective separation of gases such as hydrogen and oxygen. However, dense membranes have limited industrial application due to their low permeability compared to porous inorganic membranes. Therefore, today's commercial inorganic membrane market is dominated by porous membranes.

Metal Membrane

Dense, metal membranes are being considered for the separation of hydrogen from gas mixtures. Palladium (Pd) and its alloys are the dominant material used, due to its high solubility and permeability for hydrogen. Palladium, however, is expensive. One alternative is to coat a thin layer of palladium on a tantalum or vanadium support film. Alternative to palladium and less expensive are tantalum and vanadium, which are also quite permeable to hydrogen.

Recent focus is on supported thin metallic membranes with thickness ranging from submicron to a few ten microns. The advantages include reduced material costs, improved mechanical strength and possibly higher flux. The main

developments have been the production of composite palladium membranes for use in catalytic membrane reactors (CMRs). This development is based on the concept of process intensification, one important aspect if which is the potential for combining the reaction and separation stages of a process in one unit. One such application is the CMR. Apart from the benefit inherent in cost reduction of plant and maintenance, there is also the potential attainment of higherconversions and product yields.

The composite palladium membrane used in the CMR is composed of a thin layer of Pd, or Pd alloy, deposited onto a porous substrate, such as a ceramic or stainless steel.

The composite palladium membrane is placed adjacent to a catalyst bed and effects the selective removal of hydrogen from the catalytic reaction source. Another application is the use of these membranes to control the feed rate during partial oxidation reactions (e.g. addition of hydrogen).

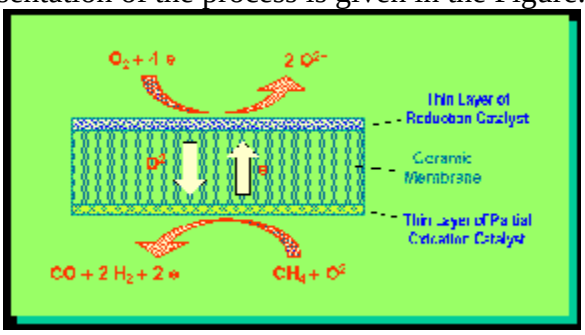
A major problem associated with metal membranes is the surface poisoning effects (e.g. by a carbon-containing source) which can be more significant for thin metal membranes.

Ceramic Membrane

These membranes are made from aluminum, titanium or silica oxides. They have the advantages of being chemically inert and stable at high temperatures. This stability makes ceramic microfiltration and ultrafiltration membranes particularly suitable for food, biotechnology and pharmaceutical applications in which membranes require repeated steam sterilization and chemical cleaning. Ceramic membranes have also been proposed for gas separations.

An example application of recent development is in the production and processing of syngas (synthetic gas - a mixture of hydrogen and carbon monoxide). The key part of the process involves the separation of oxygen fromair in the form of ions to oxidize the methane.

A schematic representation of the process is given in the Figure.



Oxygen feeding from air is split at the perovskite-type membrane surface and is transported as O^{2-}. The advantage of the membrane-based process is that the production of syngas takes place in a single-step operation occurring on one of the membrane sides, This process eliminates the need for a separate oxygen production plant, and might lead to significantly lower energy and capital costs.

Some remaining problems include:

- Difficulties in proper sealing of the membranes in modules operating at high temperature
- Extremely high sensitivity of membranes to temperature gradient, leading to membrane cracking
- Chemical instability of some perovskite-type materials in the high temperature reduction conditions

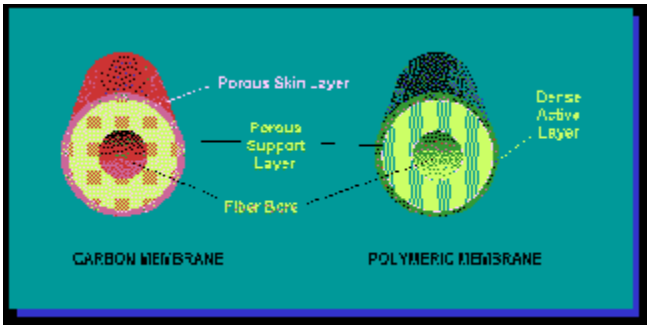
Carbon Membranes

Carbon molecular sieve (CMS) membranes have been identified as very promising candidates for gas separation, both in terms of separation properties and stability. Carbon molecular sieves are porous solids that contain constricted apertures that

approach the molecular dimensions of diffusing gas molecules. As such, molecules with only slight differences in size can be effectively separated through molecular sieving.

Carbon membranes can be divided into 2 categories: supported and unsupported. Unsupported membranes have 3 different configurations: flat (film), hollow fiber and capillary while supported membranes consisted of 2 configurations: flat and tube.

The Figure showed a comparison between carbon asymmetric membrane and polymeric asymmetric membrane where the main difference is in the skin layer.



CMS membranes can be obtained by pyrolysis of many thermosetting polymers such as poly(vinylidene chloride) or PVDC, poly(furfural alcohol) or PFA, cellulose triacetate, polyacrylonitrile or PAN, and phenol formaldehyde.

Zeolite Membranes

Zeolites are microporous crystalline alumina-silicate with a uniform pore size. Zeolites are used as catalysts or adsorbents in a form of micron or submicron- sized crystallites embedded in millimeter-sized granules. The zeolite type prepared most often as a membrane is MFI.

Main problem - relatively low gas fluxes compared to other inorganic membranes …. Due to the fact that relatively thick zeolite layers are necessary to get a pinhole-free and crack-free zeolite layer. Overcome: use thin layer supported on others.

Other problem: thermal effect of zeolites. The zeolite layer can exhibit negative thermal expansion, i.e. in the high temperature region the zeolite layer shrinks …. But the support continuously expands, resulting in thermal stress problems for the attachment of the zeolite layer to the support, as well as for the connection of the individual micro-crystals within the zeolite layer.

MEMBRANE PROPERTIES

Various membrane properties can be classified as follows:

Physical - porosity, pore size and pore distribution, thickness, tortousity, thermal stability, etc.

Separation - permeate flow rate or permeation flux, permeability, selectivity.

Surface and electrochemical - streaming potential, zeta potential (or electrokinetic potential), membrane potential (voltage potential difference), surface charge density, wettability (hydrophilic or hydrophobic), etc

Electrical - impedance measurements to determine the membrane conductance and capacitance

Separation properties of membranes depend not only on their physical properties, but also on their surface charge and electrical properties. Many of these properties are inter-related, and vary from membrane to membrane, as well as theenvironment in which the membrane is exposed, e.g. feed conditions (pH, temperature, concentration of solutes, various properties of solutes, etc). Suffice to say, each membrane-solute system is unique, and must be evaluated experimentally.

Membrane Characterization

Membrane processes can cover a wide range of separation problems with aspecific membrane being required for every problem. Membranes may differ significantly in their structure and consequently in their functionality. To know what membrane to use in a particular separation process, different membranes must be characterized in terms of structure and mass transport properties. Because very different membranes are used, different techniques are required for characterization.

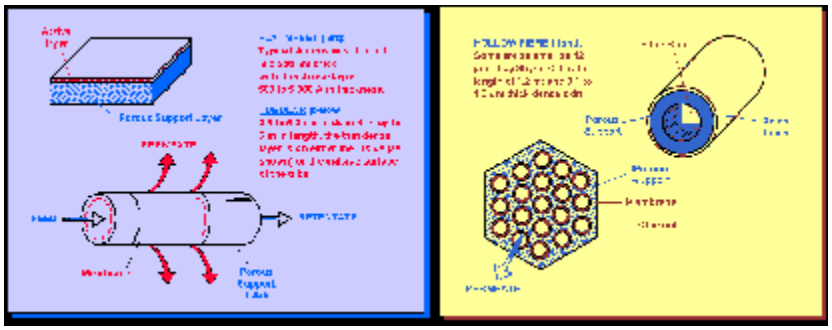
Membrane characterization is a very important part of membrane research and development because the design of membrane processes and systems depends on reliable data relating to membrane properties.

The following sections briefly discuss the following:

Characterization of Porous Membranes Characterization of Dense, Homogeneous Membranes Characterization of Charged Membranes.

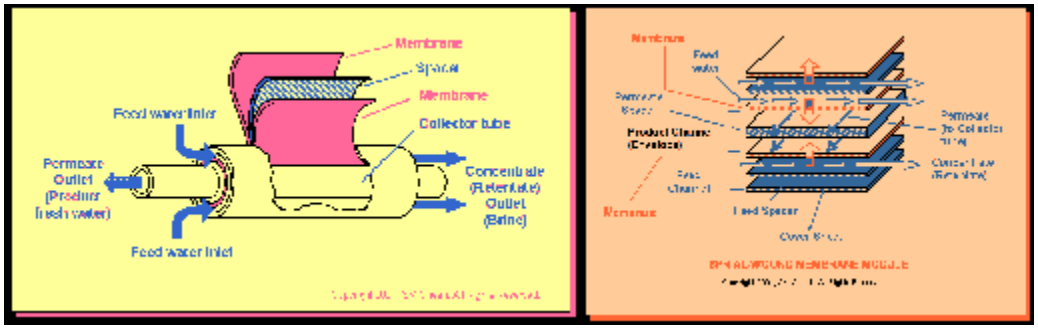
Membrane Shapes

Membranes are configured for the most part in 3 ways: (1) as **long cylinders** such as hollow fibres, capillaries or tubes, (2) as **sheets** which are either rolled up or maintained in a flat condition, and (3) as various **monolithic** designs. Common membrane shapes include flat sheet, tubular (Figure - left), hollow fiber and monolith (Figure - right).



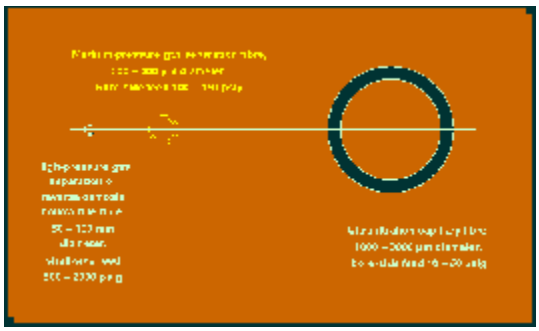
Flat sheets used in **plate-and-frame modules** can be circular, square, or rectangle in cross-section. The sheets are separated by support plates that channel the permeate.

Flat sheets can also be fabricated into **spiral-wound modules**. The simplestdesign is the **single-leaf** spiral wound module. See the Figure (left). A "leaf" or laminate, consisting of 2 membrane sheets separated by a spacer sheet forms the channel for flow of feed or permeate. The leaf is attached to the axial collector tube and is wound around the tube. The configuration is sometimes described as amembrane "envelope". Some design can have 2 or more leaves. Permeate flows through the membrane into the product channel where it is conducted spirally to the collector tube. See the Figure (right) for the flow details.



An alternative to the flat sheet membranes is the **hollow-fibre membranes**. The diameter of hollow fibres varies over a wide range from 50 to 3,000 micrometer. Fibres can be made with a uniformly dense structure, but preferably are formed as

a microporous structure having a dense selective layer on either the outside.(sometimes referred to as the shell side) or the inside surface.The Figure showed the various hollow-fibre membranes:



The dense surface layer can be either integral with the fibre or a separate layer coated onto the porous support fibre. Many fibres can be packed into bundles and potted into tubes to form a membrane module.

Fibres of 50 - 200 micrometer diameter are usually called **hollow fine fibres**. Such fibres can withstand very high hydrostatic pressures applied from the outside (shell side), so they are used in reverse osmosis or high-pressure gas separation applications (up to 1,000 psi). The feed fluid is applied to the outsideof the fibre and the permeate is removed down the fibre bore.

When the fibre diameter is 200 - 500 micrometer, the fluid is commonly applied to the inside bore of the fibre, and the permeate is removed from the outside. Thus method is used for low-pressure gas separations and for ultrafiltration or hemodialysis. Fibres with diameter greater than 500 micrometer are called **capillary fibres**.

Industrial Membrane Modules

Industrial membrane plants often require hundreds to thousands of square meters of membrane to perform the separation required on a useful scale. There are several ways to economically and efficiently package membranes to provide a large surface area for effective separation.

From an overall cost standpoint, not only is the cost of membranes per unit area important, but also the cost of the containment vessel into which they are mounted. Basically, the problem is how one can pack the most area of membranesinto the least volume, to minimize the cost of the containment vessel consistent with providing acceptable flow hydrodynamics in the vessel.

These packages are called membrane modules. The most important were: Plate-and-frame
Tubular
Spiral-
wound
Hollow fiber

Plate-and-Frame Modules

The plate-and-frame modules were one of the earliest types of membrane system, but because of their relatively high cost they have been largely replaced in most applications by spiral-wound modules and hollow-fiber modules. Plate-and-frame modules are now used only in electrodialysis and pervaporation systems and in a limited number of reverse osmosis and ultrafiltration applications with highly fouling conditions.

Tubular Modules

These modules are now generally limited to ultrafiltration applications, for which the benefit of resistance to membrane fouling outweighs the high cost. Tubular membranes contains as many as 5 to 7 smaller tubes, each 0.5 to 1.0 cm in diameter, nested inside a single larger tube. In a typical tubular membrane system, a large number of tubes are manifolded in series. The permeate is removed from each tube and sent to a permeate collection header. An example is shown in the Figure.

Spiral-wound modules

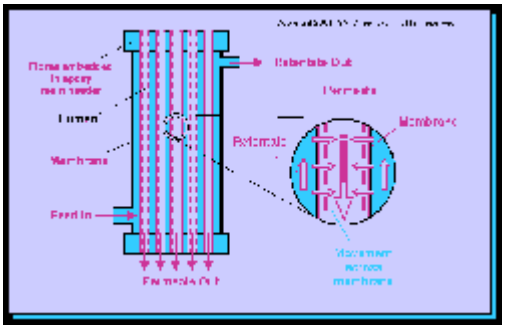
Industrial-scale modules contain several membrane envelopes, each with an area of 1 to 2 m^2, wrapped around the central collection pipe. Multi-envelope designs minimise the pressure drop encountered by the permeate travelling toward the central pipe. The standard industrial spiral-wound module is 8-inch in diameter and 40-inch long. An example of the 4-envelope (4-leaf) module is shown in the Figure. The module is placed inside a tubular pressure vessel. The feed solution passes across the membrane surface, and a portion of the feed permeates into the membrane envelope, where it spirals towards the centre and exits through the collection tube as shown in the Figure.

4 to 6 spiral-wound membrane modules are normally connected in series inside a

single pressure vessel. A typical 8-inch diameter tube containing 6 modules has 100 to 200 m^2 of membrane area. An example of a membrane tube with 2 modules is shown in the Figure.

Hollow-fibre Modules

Hollow-fibre modules are characteristically 4-8 inch (10-20 cm) in diameter and 3-5 (1.0-1.6 m) feet long. Hollow-fibre units are almost always run with the feed stream on the outside of the fibre. Water passes through the membrane into the inside or "lumen" of the fibre. A number of hollow-fibres are collected together and "potted" in an epoxy resin at both ends and installed into an outer shell. An example is shown in the Figure.



Hollow-fibre membrane modules are formed in 2 basic geometries: (a) shell-side feed design, and (b) bore-side feed design, as shown in the Figure.

In the shell-side feed design, a loop or closed bundle of fibres is contained in a pressure vessel. The system is pressurised from the shell side, and the permeate passes through the fibre wall and exits through the open fibre ends. This design is easy to fabricate and allows very large membrane areas to be contained in an economical system. Because the fibre wall must support considerable hydrostatic pressure, the fibres usually have small diameters and thick walls, typically 50- mm inner diameter and 100 - 200 mm outer diameter.

In the bore-side feed design, the fibres are open at both ends and the feed is circulated through the bore (annulus area) of the fibres. To minimise pressure drop inside the fibres, the diameter are usually larger than those of the fine fibres used in the shell-side feed design.

These so-called capillary fibres are used in ultrafiltration, pervaporation, and some low to medium pressure gas applications.

The single greatest advantage of hollow-fibre modules is the ability to pack a

very large membrane area into a single module. For example, in an 8-inchdiameter, 40-inch long spiral-wound module would contain about 20 - 40 m^2 of membrane area. The equivalent hollow-fibre module filled with fibres of 100-mmdiameter, will contain approximately 600 m^2 of membrane area.

Medical Applications of Membranes

Polymeric membranes have become widely used as components of medical devices and implants, drug delivery systems, diagnostic assays, biosensors, etc. Membrane processes are used effectively for treatment of patients with various pathologies for the removal of toxins from blood (e.g. hemodialysis) or for gas exchange with blood (e.g. blood oxygenation). Membranes of suitable molecular mass cut-off are used in bio-artificial liver, bio-artificial pancreas, and as selective barriers to prevent the immune system components from coming into contact with implants while allowing nutrients and metabolites to permeate freely to and from cells.

In medical devices, membranes exhibit in general the same properties they present in microfiltration and ultrafiltration processes.

Examples of medical applications of membranes

This section briefly discusses several applications, namely the following:

Hemodialysis (Artificial Kidney)

Blood Oxygenation (Artificial Lung)

Artificial Pancreas

Controlled Drug Delivery (Controlled-Release Pharmaceuticals)

WHOLE BROTH PROCESSING

The concept of recovering a metabolite directly from an unfiltered fermentation broth is of considerable interest because of its simplicity, the reduction in process stages and the potential cost savings. It may also be possible to remove the desired fermentation product continuously from a broth during fermentation so that inhibitory effects due to product formation and product degradation can be minimized throughout the production phase (Roffler et al., 1984; Diaz, 1988).

Bartels et al. (1958) developed a process for adsorption of streptomycin on to a series of cationic ion-exchange resin columns directly from the fermentationbroth, which had only been screened to remove large particles so that the columns would not become blocked. This procedure could only be used as abatch process.

Belter et al. (1973) developed a similar process for the recovery of novobiocin. The harvested broth was first filtered through a vibrating screen to remove large particles. The broth was then fed into a continuous series of well-mixed resin columns fitted with screens to retain the resin particles, plus the absorbed novobiocin, but allow the streptomycete filaments plus other small particulate matter to pass through. The first resin column was removed from the extraction line after a predetermined time and eluted with methanolic ammonium chloride to recover the novobiocin.

Karr et al. (1980) developed a reciprocating plate extraction column (Fig. 10.33) to use for whole broth processing of a broth containing 1.4 g dm"3 of a slightly soluble organic compound and 4% undissolved solids provided that chloroform or methylene chloride were used for extraction. Methyl-iso-butyl ketone, diethyl ketone and iso-propyl acetate were shown to be more efficient solvents than chloroform for extracting the active compound, but they presented problems since they also extracted impurities from the mycelia, making it necessary to filter the broth before beginning the solvent extraction. Considerable economies were claimed in a comparison with a process using a Pod-bielniak extractor, in investment, maintenance costs, solvent usage and power costs but there was no significant difference in operating labour costs.

An alternative approach is to remove the metabolite continuously from the broth during the fermentation. Cycloheximide production by Streptomyces griseus has been shown to be affected by its own feed-back regulation (Kominek, 1975). Wang et al. (1981) have tested two techniques at laboratory scale for improving production of cycloheximide. In a dialysis method (Fig. 10.34), methylene chloride was circulated in a dialysis tubing loop which passed through a 10 dm"3 fermenter. Cycloheximide in the fermentation broth was extracted into the methylene chloride. It was shown that the product yield could be almost doubled by this dialysis-solvent extraction method to over 1200 fig cm"3 as compared with a control yield of approximately 700 fig cm"3. In a resin method, sterile

beads of XAD-7, an acrylic resin, as dispersed beads or beads wrapped inan ultrafiltration membrane, were put in fermenters 48 hours after inoculation. Some of the cycloheximide formed in the broth is absorbed by the resin. Recovery of the antibiotic from the resin is achieved by solvents or by changing the temperature or pH. When assayed after harvesting, the control (without resin) had a bioactivity of 750 fig cm"3. Readings of total bioactivity (from beads and broth) for the bead treatment and the membrane-wrapped bead treatments

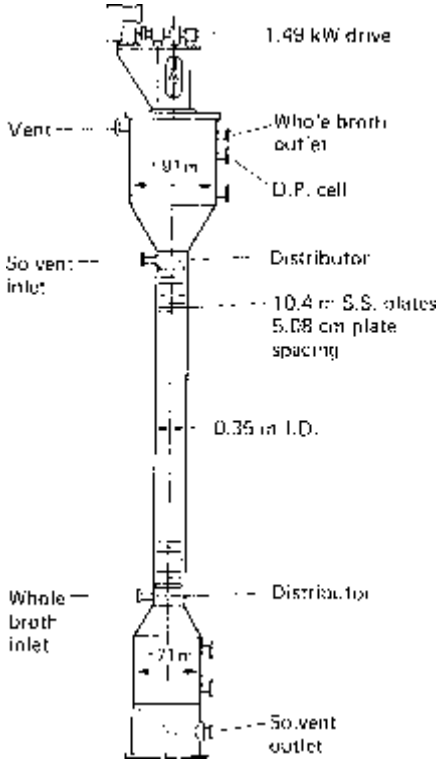


Fig. . Diagram of a 0.35-m internal diameter reciprocating plate column (Karr et al, 1980).

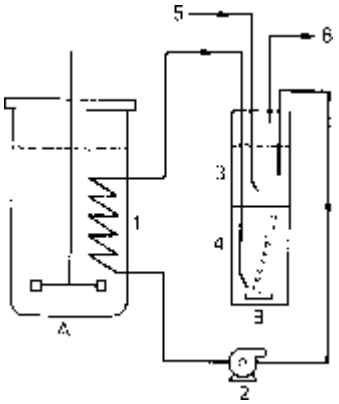


Fig. Dialysis-extraction fermentation system (Wang et al 1981).

A Fermenter BExtractor

1 Dialysis tubing

2 Pump Aqueous layer Solvent laysr Air inlet Air outlet

Fig. 10.33a. Diagram of a 0.35-m internal diameter reciprocating plate column (Karr et al, 1980).

Fig. 10.34. Dialysis-extraction fermentation system (Wang et al 1981).

(a) Vacuum and flash fermentations for the direct recovery of ethanol from fermentation broths.

(b) Extractive fermentation (liquid-liquid and two-phase aqueous) for the recovery of ethanol, organic acids and toxin produced by Clostridium tetani.

(c) Adsorption for the recovery of ethanol and cy-cloheximide.

(d) Ion-exchange in the extraction of salicylic acid and antibiotics.

(e) Dialysis fermentation in the selective recovery of lactic acid, salicylic acid and cycloheximide.

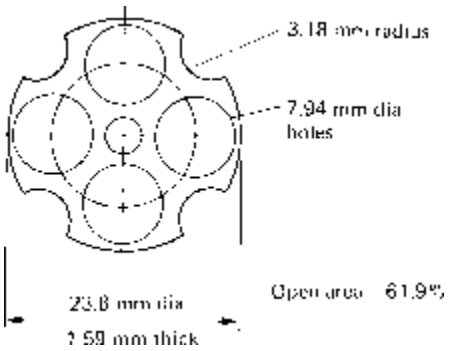


Fig. Plan of a 23.8-m stainless-steel plate for a 25-mm diameter reciprocating plate test column (Karr et al., 1980).

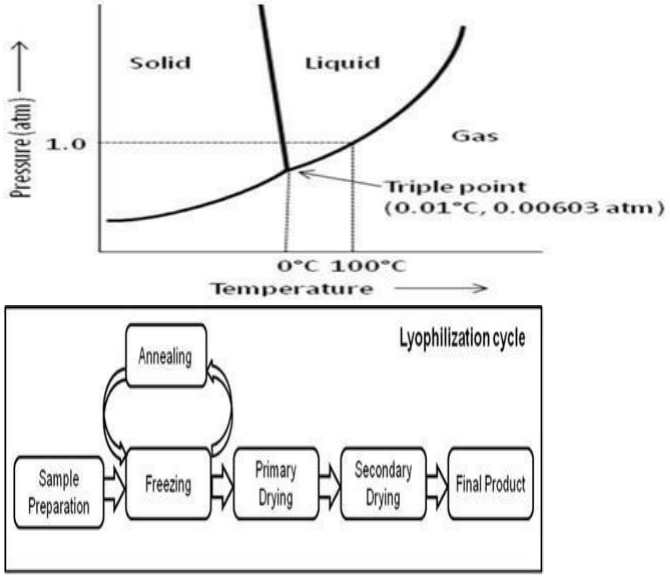
Roffler et al. (1984) reviewed the use of a number of techniques for the in-situ recovery of fermentation products:

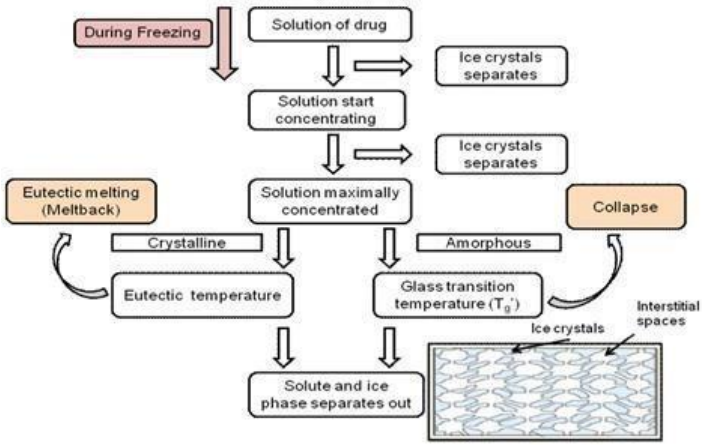
Hansson et al. (1994) have used an expanded adsorption bed for the recovery of a recombinant protein produced by E. coli directly from the fermentation broth. The protein was produced in high yields (550 mg dm"3) and > 90% recovery together with concentration (volume reduction) and removal of cells was achieved on the expanded bed. Affinity chromatography was used for further purification, and again an overall yield of > 90% obtained.

Lyophilization process is based on the principle of sublimation of ice, without entering the liquid phase. The phase diagram of water (Figure 1) represent that two phases coexist along a line under the given conditions of temperature and pressure, while at the triple point all three phases coexist.

This process is performed at temperature and pressure conditions below the triple point, to facilitate sublimation of ice. The entire process is performed at low temperature and pressure, so that useful for drying of thermolabile compounds.

various important Steps involved in lyophilization process which start from sample preparation followed by freezing, primary drying and secondary drying,to obtain the final dried product with desired moisture content (Figure 2).





The concentration gradient of water vapour between the drying front and condenser is the driving force for removal of water during lyophilization. The vapour pressure of water increases with an increase in temperature during the primary drying.Therefore, primary drying temperature should be kept as high as possible, but belowthe critical process temperature, to avoid a loss of cake structure. This critical process temperature is the collapse temperature for amorphous substance, or eutectic melt for the crystalline

substance. During freezing, ice crystals start separating out until the solution becomes maximally concentrated. On further cooling, phase separation of the solute and ice takes place. If the solute separates out in crystalline form, it is known as the eutectic temperature. In contrast, if an amorphous form is formed, the temperature is referred to as the glass transition temperature (Tg‘‘).

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SCHOOL OF BIOCHEMICAL ENGINEERING

DEPARTMENT OF CHEMICAL ENGINEERING

UNIT-IV IONIC SEPERATION SCHA3005

Electrophoresis:

It is defined as the migration of charged particles under the influence of electric field Jiselius in 1937popularised electrophoresis which become very useful analytical tool in biochemical technique.

It is relatively rapid and convenient technique capable of analyzing and purifying several different types of molecules especially proteins and nucleic aids.

Principle

It is a molecular reparation technique reparation technique that involves the use of high voltage electric current for inducing the movement of charged molecules like protein, DNA, nucleic acid in a supporting medium.

Any charged ion or molecule migrates when placed in electric field.

The rate of migration of compound depends on the net charge, size, shape and applied current.

This can be represented by the following equation

$$V = \frac{E \times q}{f}$$

Where V = velocity of migration of the particle

E = electric field in volt

q = net charge of the molecule

f = mass and shape of the molecule

Electrophoretic mobility in gels

The movement of charged molecule is called mobility (μ). The mobility of molecule towards the opposite charge for instance the protein molecule with a positive charge move towards the negative pole of the supporting medium.

The medium may be a paper, gel or a capillary tube. The movement of this charged molecule is often expressed in terms of electrophoretic

$$\mu = \frac{Exq}{Exf}$$

$$\mu = \frac{q}{f}$$

The rate of migration is directly proportional to the charge and size and shape of the molecule with different charge/mass ratio migrate under the influence of electric field at different rate and hence gets separated.

Types of Electrophoresis

Paper electrophoresis is very useful in study of normal and abnormal plasma.

- The equipment required for electrophoresis consist two units, a power pack and an electrophoresis.
- The serum is mixed with bramophenol blue a spotted at the centre of a str9p of a specially.
- When an electric field of proper voltage is pass through the paper charged protein fraction bearing different charges move at different rate.
- After a run of 5-6 hours it is dried and observed. In human serum five different band can be identified on paper electrophoresis.
- They are in the order of albumin, α_1 – globulin, α_2-globulin, β-globulin and γ-globulin.
- Albumin being the fastest moving fraction on the protein of plasma forms the last band of the paper.
- γ-globulin which is the slow moving protein forms a band on the other end.
- The rest of the protein takes their position in between these -2 bands.

Cellulose acetate paper

- They are commercially available as stripped which are thin with uniform micropore structure
- The are less hydrophile and thus hold title buffer which results in better resolution in a shorter time
- This paper shows minimum absorption and give a dear separation with distinct zone.
- Hence the compound can be easily eluted with good recovery and the process is fast completed within 1 hour.
- Other advantages are:

The strips are chemically pure and do not contain lignin hemicellulose.

Polyacrylamide gel electrophoresis (SDS-PAGE)

Electrophoresis in acrylamide gel is frequently referred to SDS PAGE (sodium diodecylsulphate polyacrylamide gel electrophoresis). The components used in PAGE are

acrylamide, bisacrylamide, TEMED (tetra methyl ethylene diamde). Cross linked polyacrylamide gel are formed from the polymerization of acrylamide monomer in the presence of small amount of bisacrylamide. Several proteins of biological importance contain more than one polypeptide chain. These proteins are referred to as oligomeric proteins.

The structure of these proteins is stabilized by hydrogen bond, disulphate band, disulphide linkage, hydrophobia interaction,mercapto ethanol breaks disulphide bridges present in the oligomericproteibn.

SDS is anionic detergent which disruput macromolecule whose structure has been stabilized by hydrophobic interaction.

SDS bindings imparts a large negative charge to denature protein.

PAGE combined with SDS is most widely used method for analyzing protein mixture quantitatively.

It is particularly useful for monitoring protein purification.

The molecular weight of most protein could be determined by measuring the mobility of most protein in polyacrylamide gel containing SDS.

Sample to be separated is boiled for 5 mins in a sample buffer containing SDS and mercapto ethanol.

This treatment completely denature protein present it the sample and imparts negative charge to the polypeptide chain.

The sample buffer also contains a tracking dye which is to monitor the electrophoretic tank.

Process

In SDS-PAGE, the gel column consist of 2 parts (stacking gel and separating gel).

The separating gel is poured into a glass tube and allows to set. Then stacking gel is poured at the top of the separating gel. The stacking gel has large pore size and separating gel has comparatively small pore size.

Samples of protein known and unknown molwt are layered on top of each column separately.

The stacking gel allows the protein to move free and concentrate over the separating gel under the influence of electric field.

Protein continues the movement towards anode. Therefore protein have same charge. All proteins travel with same mobility. However it passes through separating gel, the protein separate owing to molecular sieving properties of gel. The small protein move fast as they can pass through the pour of the gel but large protein move slowly.

A plot of distance migrated vs log of the molecular weight gives the straight line hence if the protein of unknown molecular weight is electrophoresed with 2 or more protein of known molecular weight, the molecular weight of unknown protein can be calculated with an accurately ranging from 90 – 95%.This is the most common way of estimating the

molecular weight of molecular subunit.

Applications

Fine resolution of protein mixture is possible.

Pore size can be controlled and separation depends upon the size shape and charge of the molecule.

Highly suitable for histochemical and quantitative analysis.

The method of separating protein according to the isoelectric point in a pH gradient is called electric focusing.

This method has high resolution power because ordinary paper electrophoresis resolve focusing resolve into 40 band.

In conventional electrophoresis, the pH between anode and cathode is constant.

The positive charge ion migrate towards cathode and the negative charge ions migrate towards anode.

In isoelectric focusing a stable pH gradient is arranged. The pH gradually increases from anode to cathode.

When a protein is introduced at a pH which is lower than its ionic point, it will possess a net positive charge and migrate in the direction of cathode.

One to the presence of pH gradient, the net charge of the molecule encounters a pH where net charge is zero, it will stop migrating.

This is the isoelectric point of protein. Each protein present in the mixture migrate to its isoelectric point and stops migration at that point.

Thus once the final stable focusing is reached, the resolution will be retained for longer time.

Applications

It is widely used for separation and identification of serum protein.

It is widely used in food and agriculture industry, forensic and genetic lab.

It is used in the research for reading enzymology, immunology, biochemistry, etc.

A standard gel electrophoresis provide huge advantage for molecular biology research. However it was unable to separate large molecules of DNA effectively.

This technique of pulse gel electrophoresis is used for the separation of large DNA molecules more than 15 – 20 kb.

Principle

Electric field is not constant and changes repeatedly pulse in direction and strength during the separation such that DNA molecular are forced to continuously orient and reorient themselves.

Larger the DNA molecules, longer is the time required for its orientation.

Process

The process is relatively similar to performing a standard gel electrophoresis except instead of constantly running the voltage in one direction, the voltage is periodically fixed among 3 directions that one4 runs in the central axis and other at the angel of 60^o either side.

This electric abrupt pulse changes the conformation of DNA molecules. They would be oriented by the influence of current in one direction and then reorient as a new electric field at a different angle to the first one was turned on.

DNA molecules tends to shutter through the gel matrix and the gel becomes fluidly during the electrical pulsing.

The pulse time are equal to each direction resulting in a net forward direction by DNA.

This process also ensures the migration of DNA on absolutely straight trace. The process was easier to dissolve DNA fragment upto 2000 kB with good resolution.

Applications

i) It is significant tool for characterization of larger molecules.

ii) The pore size can be controlled and the separation depends upon the size, shape and charge of the molecule.

iii) It is suitable for histochemical and quantitative analysis.

Agarose gel

Agarose is a natural product purified from the seaweed Rhodophyta.

It is a linear polysaccharide, agarose dissolve well and boiling and forms a gel when cooled.

1-3% of agarosegel are prepared by suspending dry agarose in a aqueous buffer (tris acetate buffer, tris buffer) and then boiling the mixture allowed to under a room temperature to form a rigid gel.

The pore size is determined by the concentration of agarose.

Low concentration produces larger pore size while high concentration produces small pore size.

Agarose gel are mostly used to study DNA molecules having high molecular weight which cannot penetrate through polyacrylamide gel but can penetrate through 0.8% of agarose.

Procedure

Agarose gel are casted by melting the agarose in the presence of a desired buffer until a clean transparent solution is achieved.

The melted solution is then powered into a mould and allowed to

harden. Upon hardening, the agarose forms a matrix and the

components are separated.

The gel is connected to a anode and cathode to the buffer reservoir through the paper.

Applications

1. It is used to isolate large number of protein and identity the molecular weight and purity.
2. It is convenient method to study DNA / RNA fragment.
3. The sequence of the DNA and molecular weight can be determined.
4. The location of a DNA in the gel can be directly determined by the use of fluorescent dye like ethidium bromide.

System Concept

The chemical analysis of blood and other body fluids is one of the earliest forms of diagnostic criteria leading to the investigations of the diseases.

In the early years of clinical biochemistry most of the required analyse were performed on urine since it available easily in large quantities.

With the development of semimicro and micro analytical techniques, analysis can be carried out with minute quantities of samples.

Therefore, the analysis of the blood serum (or) plasma is becoming more frequent. A majority of the blood analysis can now be performed by an automated system.

Advantages of Autochemical analysis

1. Precision (increased)
2. Minute quantities of samples are enough for analysis.
3. Repetitive monotonous work load of humans can be avoided and hence the error.
4. More reliable and accurate.

Types of analysers

1. **Electrolytic analysis**: It includes the detection of ions such as potassium, sodium, chloride and bicarbonate ions.
2. **Protein analysis**: It detects the presence of proteins, biolirubins (enzyme secreted by bilejuices), alkaline phosphatase.

Steps involved in automated analysis

1. Specimen identification
2. Specimen transport and delivery
3. Removal of proton and other interfering components.
4. Reagent handling and delivery
5. Chemical reaction phase

6. Measurement procedure
7. Signal processing and data handling
8. Report preparation.

These steps are carried out sequentially under the control of microprocessors.

System details

The automated system is usually a continuous, flow system, in which individual operations are performed on the flowing stream as it moves through the system.

The end product passes through the colorimeter to measure concentrations of various constituent. The final results are recorded on a strip chart recorder along with a calibration curve. So that the concentration of the unknowns can be calculated.

The automated system consists of a group of modular instruments.

1. Sampling unit
2. Proportioning pump
3. Manifold
4. Dialyser
5. Heating bath
6. Colorieter (flame photometer /fluorometer)
7. Recorder
8. Function monitor

System components

Sampling unit: The sampling unit enables to an operation to introduce unmeasured samples and standards into the autoanalyser system.

It consists of a circular turn table carrying around its rim 40 disposable polystyrene cups of 2 ml capacity.

The sample plate carrying these cups rotates at a predetermined speed.

The movement of the turntable is synchronized with the movements of a sampling

crook. The hinged tubular crook is fitted at a corner of the base.

The crook carries a thin flexible polythene tube which can dip into a cup and allow the contents water, standard (or) test solution to be aspirated. At regular inter volts, the crook is raised, so that the end of the sample tube is lifted clear of the cup.

Between each sampling, the crook enters a receptacle of water (or) suitable wash fluid to reduce cross – contamination of one sample with another.

The plate then rotates a distance sufficient to allow the tube, when it next moves down to dip into the next cup.

One complete rotation of the plate thus presents 40 samples. Volume of the sample

ranges from 0.2 to 1.0 ml.

In new version, thus successive samples are separated by a column of water instead of air. This provides a better separation.

Sample size : 0.1 to 8.5 ml

A programming can will help in rotational movement of the sampler.

Proportioning pump

The function of the proportioning pump is to continuously and simultaneously push fluids, air and gases through the analytical chain.

It is the heart of the automatic analysis system.

Samples and reagent stream is are driven by a single peristaltic pump which consists of two parallel stainless steel roller chains.

A series of flexible plastic tubes, one from the sampler, the other from reagent bottles (or) simply drawing in air is placed length wire along the platen.

The roller head assembly is driven by a constant speed gear motor.

When the rollers are pressed down and the motor switched on the compress the tubes containing, the liquid stream (sample, standard and reagents) against the platen.

Roller head rotates at a constant speed proportioning pumps are available either for single speed (or) for two-speed operation.

The single speed pump has the capacitor synchronous gear head utilizing 10 rpm output shaft at 50 Hz.

The two speed has a nonsynchronous 45 rpm motor.

In two speed operation, the slow speed helps in filling the system and washing draining.

High speed is not used for analysis.

Heavy duty pump is also available, which enables 23 pumps tubes to be used simultaneously.

Tubes are stretched before run

- Constant use causes

a) Loose – elasticity

b) Reduction in pump efficiency

Manifold

A manifold mainly consists of a platter, pump tubes, coils, transmission tubings, fittings and connections.

A separate manifold is required for each determination.

The pump tubing and the connected coils are placed on a manifold platter, which

keeps them in proper order for each test.

The pumping tubing are specially made they are of premeasured length and are meant to introduce all constituents of an analysis into the system.

Physical, chemical properties of the tubing are extremely important in the correct functioning of the pump.

It must be so flexible to maintain the flow rate.

- Standard transmission tubing
- Solva flex tubing
- Acid flex tubing
- Polyethyelene tubing
- Glass tubing

Size - 0.005 – 0.110 inch

Flow rate – 0.015 µl/min -3.90 µl/min

1) Mixing coil [2 types of coils]

Mixing coil are used to mix the sample (or) reagents. As the mixture rotate through a coil, the air bubble along with the rise and fall motion produces a completing homogeneous mixture.

Mixing coils are placed in a horizontal position to permit proper mixing

2) Delay coil

- Used to delay specimen for chemical reactions
- 40 ft long 1.6 mm in diameter Volume: 28ml

Dialyser

In analytical chemistry it is necessary to remove protein cells to obtain an interference free analysis.

This is accomplished by dialysis in the autoanalysis.

The dialyser module consists of a pair of Perspex plates, which are mirror grooved in continuous channel.

A semipermeable cellophane membrane is placed between the two plates and the assembly is clamped together.

The continuous groove channel thus get divided into two halves and the dialysis occur across the membrane.

A solution containing substance to be analysed passes along one half usually the upper one of the channel, while the solvent that is receptive to the substance to the substance to be removed enter the other half.

The substance to be separated from the sample diluents stream, will diffuse through

the semipermeable membrane by armotic pressure into the recipient stream and the non-diffusable particles will be left behind.

The cellophane membrane usually used in the dialyser has a pore size of 40-60 Å. The rate of dialyser is stated to be dependent upon temp. 37^{o}C.

The temp is kept constant with a thermostatically controlled heater and a stirrer.

The channel path is 87 inch along which provides a large surface presentation to the dialysis membrane.

The plates of the dialyses most be matched set if the plates are not matched set the channels may be slightly off, causing leakage loss of dialyzing area, which would ultimately result in loss of sensitivity.

Quality of solute, concentration gradient, duration, area temperature, thickness and porosity determines the separation of protein during dialysis via semipermeable membrane.

Heating Bath

It is then passed to a heating. The heating bath is a double walled insulated vessel, in which a glass heating coil is immersed in a mineral oil. A thermostatically controlled heater maintain a constant temperature 0.1^{o}C, which can be read on thermometer.

Measurement of Techniques

Almost all automated analyser uses absorption as the major measurement.

Colorimeter:

Colorimeters used in the automated system continuously monitor the amount of light transmitted through the sample.

They employ flow through cuvettes. Eg: dual beam type colorimeter.

Recorders

The most common type of records used with automated system is the dc voltage null balance potentiometric recorder. Initially the setting of recorder pen is made to100% transmission.

Reagent blank kept running for some time unit a smooth baseline is established on the after obtaining a good baseline the run can be begun with a series of standard followed by the samples.

Since concentration of substance in a sample is related logarithmically to the percent transmission when this is plotted on a graph, the curve will not be linear.

Actual measurement are made only when it reaches steady state plateau.

Function monitor

Recording at steady state levels, the electrical output of the phototube is given to an oscilloscope called the function monitor.

Each sample curve is recorded entirely this enabling the operator to see all curves

for each sample at all times.

SAMAC – II

The advanced version based on the auto-analyser concept is the Samac II (sequential multiple analysis plus computer).

This is a multichannel analyser which can perform 23 simultaneous analyser on the specimen.

168 specimens can be loaded on the system thereby SAMAC carries out about 2500 test results per hour.

The system is completely computer controlled usually two host computers and 10 microprocessors.

The system has two main subsystems.

- Analytical Processor: For instructing and monitoring analysis
- Results Processor: To provide data handling of results and preparation of edited report. Approximately 600 µl of specimen is required for complete analysis. After the specimen

is sampled, diluted with distilled H_2O and segmented with air bubbles.

It is passed through a series of analytical cartridges (which contains reagent).

Each cartridge is a single channel analyser and includes peristaltic pump, pump tubes, mixing tubes and flow cells, pump tubes usually need 200 h of operation, dialyser for protein separation, heating bath for color development, spectrophotometry for quantitation for most analytes, Wavelength for each flow cell is selected through a computer, transmittance signal from the photomultiplier is converted to absorbance by a logarithmic amplifier, analogue signal is digitized in an A – D converter, Digital data are processed for peak detection, which is checked and tested for abnormal waveform, stored in memory.

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SCHOOL OF BIOCHEMICAL ENGINEERING

DEPARTMENT OF CHEMICAL ENGINEERING

UNIT-V DISTILLATION AND FOAM SEPERATION
SCHA3005

Introduction to Adsorption

Adsorption is one of the most important surface processes and its knowledge is essential for a chemical engineer due to its wide range of applications in almost all fields of science and technology.

Definition:

Adsorption is defined in many ways, some of the standard definitions of adsorptions are:

- Adsorption is the adhesion of atoms, ions, biomolecules or molecules of gas, liquid, or dissolved solids to a surface
- The process by which molecules of a substance, such as a gas or a liquid, collect on the surface of another substance
- Adsorption is the process through which a substance, originally present in one phase, is removed from that phase by accumulation at the interface between that phase and a separate (solid) phase.

Applications:

Some of the standard applications of adsorption are:

- Heterogeneous Catalysis- This is probably the most important application relevant to chemical engineering. The reaction mechanism of how a reactant reacts on a catalyst surface revolves around adsorption. Therefore designing catalyst, reactors and studying them requires knowledge of adsorption
- Separation- Adsorption is used as a separation process in many chemical as well as bio chemical industries to separate gaseous or liquid mixtures. Designing adsorption equipment like fixed bed adsorbers, gas drying, pressure swing adsorption etc. , chromatography requires knowledge of adsorption.
- Many experiments in the lab use adsorption as a process to calculate various parameters like surface concentration, porosity, change in surface energies, pore surface area etc.

NOTE: ***Adsorption must not be confused with absorption which is a bulk process in which a substance diffuses into the bulk of another substance unlike adsorption which is only a surface process.***

Adsorption Isotherms

Now let us study a bit more of the physics of adsorption. Adsorption is usually described through adsorption isotherms that is *the amount of adsorbate on the adsorbent as a function of its pressure (if gas) or concentration (if liquid) at constant temperature*. The adsorption isotherm is the equilibrium relationship between the concentration in the fluid phase and the concentration in the adsorbent particles at a given temperature. The quantity adsorbed is nearly always normalized by the mass of the adsorbent to allow comparison of different materials. Some typical adsorptions are shown in the figure below

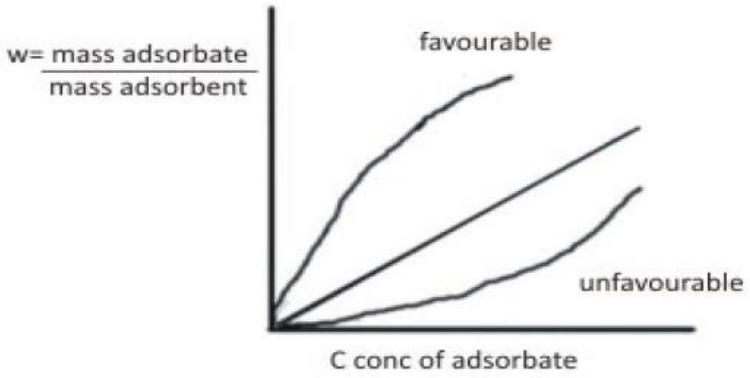


Fig. 7.2: Favourable and unfavourable adsorption

Different adsorption isotherms

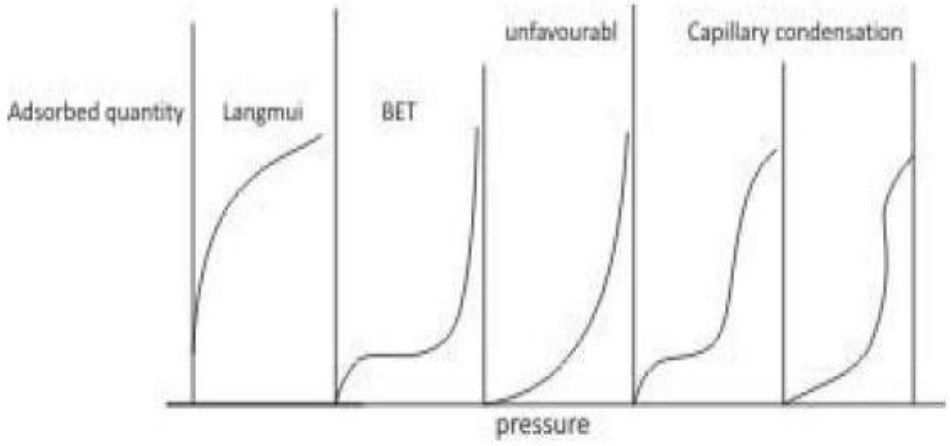


Fig. 7.3: Different adsorption isotherms

Langmuir Adsorption Isotherm

The simplest equation for adsorption under dynamic equilibrium condition was derived by Irving Langmuir in 1916. He got the Nobel Prize in 1932 in chemistry for his contributions to surface chemistry. It can be used to predict monolayer physisorption as well as chemisorption. Let us derive its equation in terms of partial pressure of the gas (at constant temperature) and the amount of adsobate adsorbed.

Assumptions

Langmuir made several assumptions in order to simplify his analysis. The main assumptions in Langmuir adsorption isotherm are

- The surface is like a checkerboard and made up of many active (adsorbing) sites (see figure below).
- Each active site can adsorb only one molecule of the adsorbate.
- There cannot be multilayer adsorption. (In other words the process is adsorption limited)
- All surface sites have constant heat of adsorption (there is no difference between any two active sites)
- Adsorbed molecules don't interact with each other on the surface. (there is no motion of adsorbed molecules on the surface)
- The surface containing the active sites is perfectly like a flat plane with no corrugations.

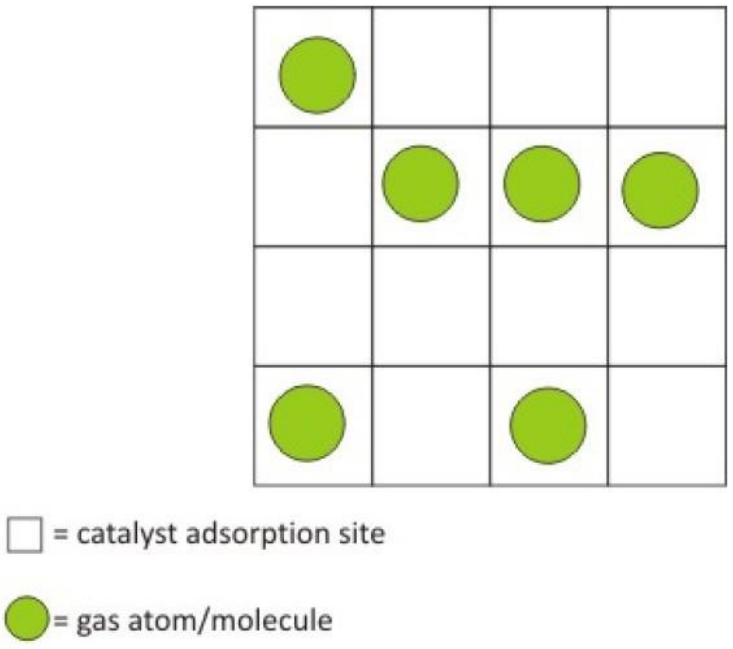


Fig. 7.4: Surface of adsorbent

According to Langmuir, the rate of adsorption depends on the following 4 factors

- The rate of collision of the adsorbate molecule of mass 'm' which is at pressure 'p' is proportional to $\frac{p}{\sqrt{2\pi mkT}}$ per unit surface area at the constant temperature.
- The activation energy E^{act} of adsorption ,since this determines the fraction of the colliding molecules possessing the necessary energy to be adsorbed (see figure 7.5)

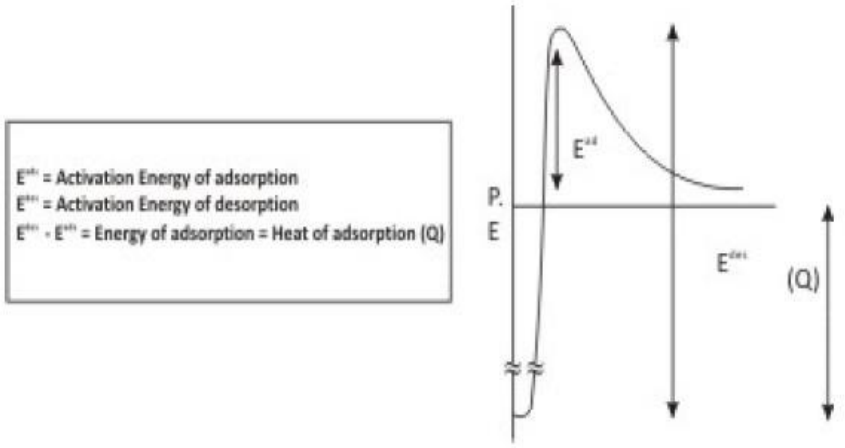


Fig. 7.5: Activation energy diagram for adsorption

- The fractional coverage of the surface $f(\theta)$ which is the amount of surface that is exposed/available for a single site adsorption $= (1-\theta)$, where

$$\theta = \text{fraction of sites covered} = \frac{number\ of\ sites\ which\ have\ absorbed\ molecules}{total\ number\ of\ sites} \quad (7.1)$$

- σ which is the 'fraction of the total number of colliding molecules which results in adsorption'.

Shortcomings of the Langmuir adsorption isotherm

- Langmuir assumed that the heat of adsorption is independent of the coverage θ which is not what is observed.
- Multilayer adsorption is not taken into account
- Lateral interactions on the surface were ignored

Non Langmuirian Adsorption Isotherms

Many corrections to the Langmuir adsorption isotherm were suggested that accounted for its shortcoming. Two of the more famous ones are

- **Freundlich isotherm**: $\theta = k.p^n$.

  It can be theoretically derived from Langmuir isotherm by assuming that heat of adsorption falls exponentially as the coverage is increased.

1. **Temkin isotherm:** $\theta = \frac{RT}{\Delta H\alpha} \ln(A_0 p)$.

   It can be derived from Langmuir isotherm assuming the heat of adsorption decreases linearly with coverage.

NOTE: Langmuir adsorption isotherm can also be derived using basic statistical mechanics principles by considering the grand canonical ensemble and the corresponding Helmholtz free energy.

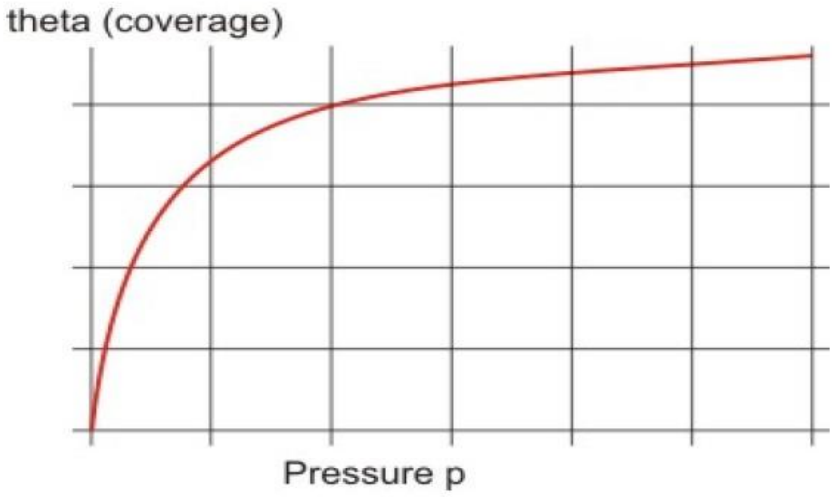


Fig. 7.6: Langmuir Adsorption isotherm

How Does Adsorption occur?

As mentioned above adsorption is a surface phenomenon. It occurs due to the imbalance of forces at the surface of a material. This lead to formation of bonds (Covalent, ionic, Van der Waals, Hydrogen bonds etc.) between the surface molecules (**adsorbents**) and the molecules in the fluid phase (**adsorbate**).

Physisorption

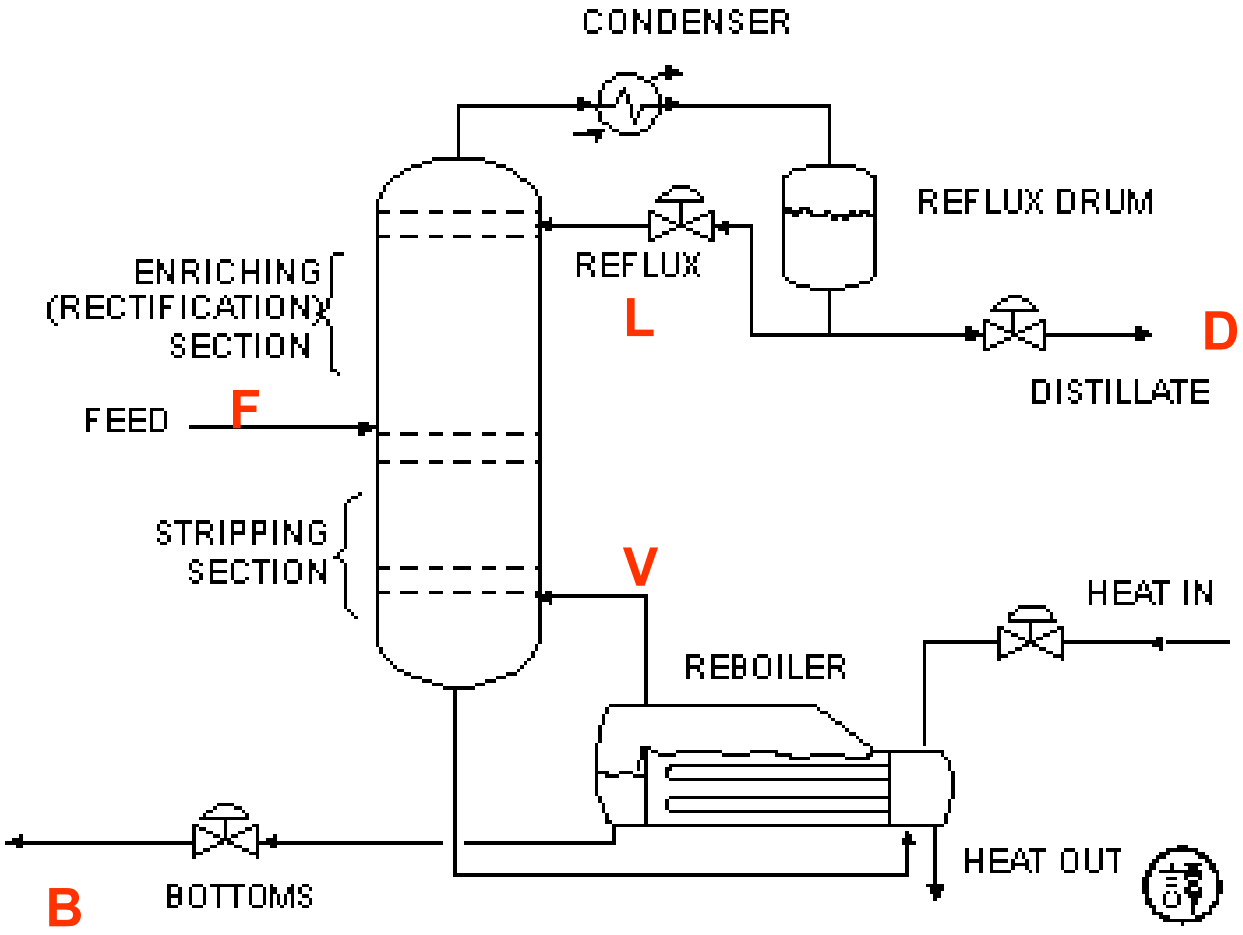
Adsorption in which the forces involved are intermolecular (i.e., van der Waals, hydrogen bonding) of the same kind as those responsible for the non-ideality of real gases and the condensation of vapours etc. , and which do not involve a significant change in the electronic orbital patterns of the species involved is called physisorption.

Chemisorption

A chemical process in which a reacting molecule forms a definite chemical bond with an unsaturated atom, or a group of atoms (an active centre) on a catalyst surface, and electron transfer is involved is known as chemisorption.

***Note*: In practice no absolutely sharp distinction can be made between chemisorption and physisorption, although generalities apply.**

Distillation



Distillation is a widely used method for separating mixtures based on differences in the conditions required to change the phase of components of the mixture. To separate a mixture of liquids, the liquid can be heated to force components, which have different boiling points, into the gas phase. The gas is then condensed back into liquid form and collected.

Repeating the process on the collected liquid to improve the purity of the product is called double distillation. Although the term is most commonly applied to liquids, the reverse process can be used to separate gases by liquefying components using changes in temperature and/or pressure.

A plant that performs distillation is called a *distillery*. The apparatus used to perform distillation is called a *still*.

Uses of Distillation

Distillation is used for many commercial processes, such as the production of gasoline, distilled water, xylene, alcohol, paraffin, kerosene, and many other liquids. Gas may be liquefied and separate. For example: nitrogen, oxygen, and argon are distilled from air.

Types of Distillation

Types of distillation include simple distillation, fractional distillation (different volatile 'fractions' are collected as they are produced), and destructive distillation (usually, a material is heated so that it decomposes into compounds for collection).

Simple Distillation

Simple distillation may be used when the boiling points of two liquids are significantly different from each other or to separate liquids from solids or nonvolatile components. In simple distillation, a mixture is heated to change the most volatile component from a liquid into vapor.

The vapor rises and passes into a condenser. Usually, the condenser is cooled (e.g., by running cold water around it)to promote condensation of the vapor, which is collected.

Steam Distillation

Steam distillation is used to separate heat-sensitive components. Steam is added to the mixture, causing some of it to vaporize. This vapor is cooled and condensed into two liquid fractions. Sometimes the fractions are collected separately, or they may have different density values, so they separate on their own. An example is steam distillation of flowers to yield essential oil and a water-based distillate.

Fractional Distillation

Fractional distillation is used when the boiling points of the components of a mixture are close to each other, as determined using Raoult's law. A fractionating column is used to separate the components used a series of distillations called rectification. In fractional distillation, a mixture is heated so vapor rises and enters the fractionating column. As the vapor cools, it condenses on the packing material of the column. The heat of rising vapor causes this liquid to vaporize again, moving it along the column and eventually yielding a higher purity sample of the more volatile component of the mixture.

Vacuum Distillation

Vacuum distillation is used to separate components that have high boiling points. Lowering the pressure of theapparatus also lowers boiling points. Otherwise, the process is similar to other forms of distillation. Vacuum distillation is particularly useful when the normal boiling point exceeds the decomposition temperature of a compound.

REMOVAL OF INSOLIBALES FOAM SEPARATION

Foam separation is a chemical process which falls into a category of separation techniques called "Adsorptive bubble separation methods". It is further divided into froth flotation and foam fractionation. A variety of materials can be concentrated as well asseparated from one another using foam separation techniques that make use of thetendency of surface-active components in a solution to preferentially concentrate at thesolution/gas interface. Nonsurface active agents that are capable of associating withsurface-active agents can also be separated using these techniques. The various anionssuch as alkyl benzyl sulfonate; chromate; cyanide and phenolate; cations of, for example, dodecylamine. mercury, lead, and strontium; proteins; microorganisms; and minerals.The attractive feature of this group of techniques is its effectiveness in the concentrationrange that is too dilute for

the successful use of most other techniques. Furthermore.these techniques are ideally suitable for also treating materials that are too sensitive tochanges in temperature.

PRECIPITATION

Ammonium sulfate precipitation is a method of protein purification by altering thesolubility of protein. Ammonium sulfate is commonly used due to its high solubility that allows salt solutions with high ionic strength. At sufficiently high ionic strength, the protein will be almost completely precipitated from the solution. Differential precipitation of proteins by ammonium sulfate is one of the most widely used preliminary purification procedures. It is based on proteins having differing solubility in ammonium sulfate solutions and can result in a two- to five-fold increase in specific activity. Provided that appropriately buffered ammonium sulfate solutions are used to protect the desired activity, recoveries approaching 100% can be expected. A typical protocol consists of adding ammonium sulfate to give specific percentage saturation, followed by a period of time for proteins to precipitate and a centrifugation step to collectthe precipitate.

SEDIMENTATION:

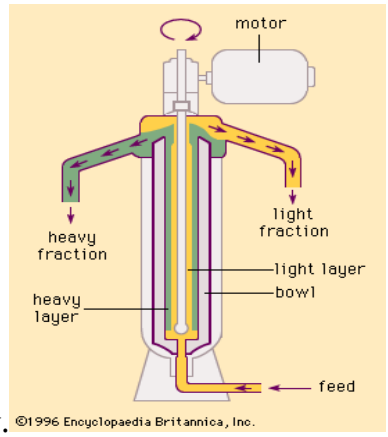
Sedimentation is the tendency for particles in suspension to settle out of the fluidin which they are entrained and come to rest against a barrier. This is due to their motion through the fluid in response to the forces acting on them: these forces can be due to gravity, centrifugal acceleration, or electromagnetism. In geology,sedimentation is often used as the opposite of erosion, i.e., the terminal end of sediment transport. In that sense, it includes the termination of transport true bedload transport. Settling is the falling of suspended particles through the liquid, whereas sedimentation is the termination of the settling process. Even small molecules supply a sufficiently strong force to produce significant sedimentation. The term is typically used in geology to describe the deposition of sediment which results in the formation of sedimentary rock, but it is also used in various chemical and environmental fields to describe the motion of often-smaller particles and molecules. This process is also used in the biotech industry to separate cells from the culture media.

CENTRIFUGATION

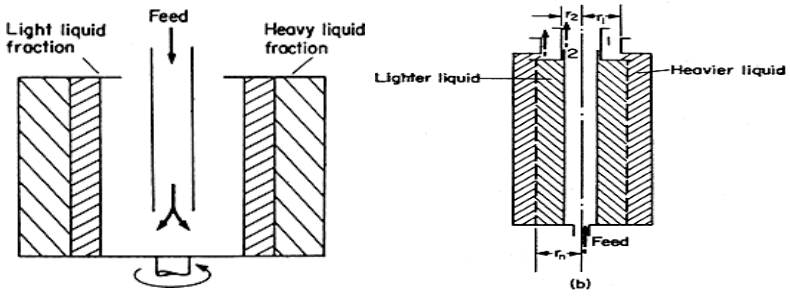
Centrifugation is a process which involves the application of the centripetal force for the sedimentation of heterogeneous mixtures with a centrifuge, and is used in industrial and laboratory settings. This process is used to separate two immiscible substances. More-dense components of the mixture migrate away from the axis of the centrifuge, while less-dense components of the mixture migrate towards the axis. Chemists and biologists may increase

the effective gravitational force on a test tube so as to more rapidly and completely cause the precipitate (pellet) to gather on the bottom of the tube.

- Feed added to spinning bowl
- Sedimentation of particles occurs in centrifugal field
- Flow is upwards at a particular rate which determines residence time in device
- Separation happens if sedimentation velocity is high enough for particle to reach side of bowl within residence time
- Large particles have higher settling velocities than small particles
- Both large and small are still particles, have small Reynolds no.s (<1) and obey Stokes‟ Law.



- **Separation of milk into skimmed milk and cream is done with a centrifuge**



Centrifugal Motion

- Centrifugal acceleration = $r\omega^2$
- ω is the angular velocity in rad/s
- r is the radius of rotation
- Centrifugal force = $mr\omega^2$
- m is the mass of the particle

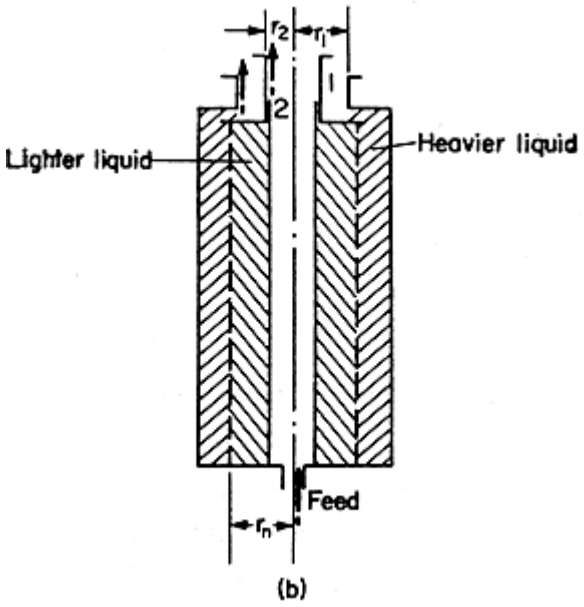
Sigma Factor

- The capacity of a centrifuge is defined by Σ
- Q is the throughput (m^3/s) at which all particles with a terminal velocity ≥

u_T (m/s) are retained

- Σ has units of m^2 and is equivalent to the cross sectional area of a thickener with the same capacity

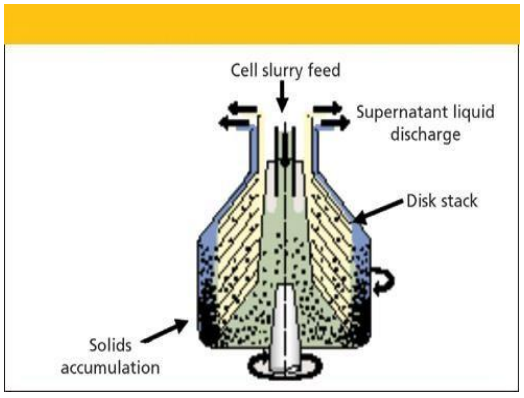
- **The contents of a fermenter are discharged to a centrifuge**
- Volume of material is 100 m^3
- Centrifugation time is 5 hrs
- Particle size is 3 □m – all particles of this size are separated
- Density of solid phase 1090 kg/m^3
- Cell free liquid density 1025 kg/m^3
- Cell free liquid viscosity 0.005 Pa.s
- **Calculate the capacity factor, Σ**



Bowl Centrifuge

Sigma Factor – Bowl Centrifuge

The Disc Stack Centrifuge



large particles have higher settling velocities than small particles Cellular debris ends up at the outer edge of the bowl

Soluble intracellular material passes through with the clarified liquid Discs give a higher sigma factor

Benefit of Discs

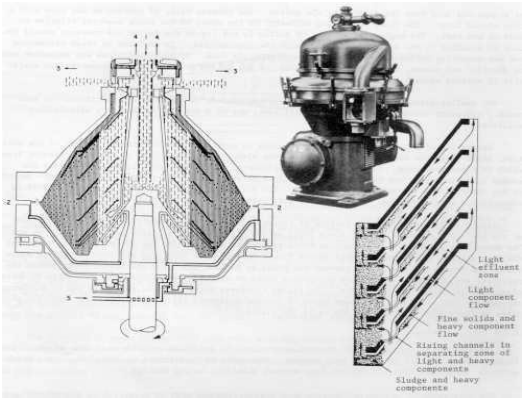
The discs split the stream into a large number of very thin layers

thereby improving separation

Solids flow downwards on bottom face ofdisc

Liquid flows upwards on top face of disc

Sigma factor no. of discs



Disc Stack Centrifuge Capacity

For the disc stack centrifuge:

ω is the angular velocity (rad/s)

n is the number of discs

R is the outer radius of the discs

(m) r is the inner radius of the discs

(m)

θ is the angle between disc and vertical (rad)

g is the acceleration due to gravity (m/s^2)

CO6 - Knows the current scenario of advanced separation process.

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