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SCHOOL OF BIO AND CHEMICAL ENGINEERING

DEPARTMENT OF CHEMICAL ENGINEERING

HEAT AND MASS TRANSFER FOR BIOLOGICAL SYSTEMS

SCHA1311

UNIT – 1 PRINCIPLES OF MASS TRANSFER- SCHA1311

UNIT 1

PRINCIPLES OF MASS TRANSFER

Introduction to Mass transfer and diffusion, Ficks law for molecular diffusion, Molecular diffusion in gases, Molecular diffusion in liquid, Molecular diffusion in solids, Miolecular diffusion in biological solutions and gels.

Introduction to convective mass transfer, Convective mass transfer coefficients. Analogy between Mass, Heat and momentum transfer. Mass Transfer and its Applications

Air is a mixture of various gases. Whenever we have a multicomponent system with a concentration gradient, one constituent of the mixture gets transported from the region of higher concentration to the region of lower concentration till the concentration gradient reduces to zero. This phenomenon of the transport of mass as a result of concentration gradient is called 'Mass Transfer'.

The mass transfer phenomenon is analogous to heat transfer phenomenon. In heat transfer - heat energy flows in a direction of decreasing temperature gradient and ceases when the temperature gradient reduces to zero. In mass transfer - the transfer of mass takes place in the direction of decreasing concentration gradient and ceases when the concentration gradient is zero.

The. common examples of mass transfer in our everyday life and in many

industries are:

- diffusion of smoke discharged by tall chimney into the atmosphere,
- a drop of ink diffusing in a glass of still water,
- evaporation of a drop of perfume in a room,
- humidification of air flowing over a spray pond or cooling tower,
- mixing of diesel or petrol with air inside an internal combustion engine,
- diffusion welding of metals,

- diffusion of neutron in a nuclear reactor.

Different Modes of Mass Transfer

There are basically two modes of mass transfer:

(i) Mass Transfer by Diffusion - the transport of mass by random molecular motion in quiescent or laminar flowing fluids is known as mass transfer by 'diffusion' and is analogous to heat transfer by conduction. Mass transfer by diffusion occurs due to (a) concentration gradient, (b) temperature gradient, and (c) hydrostatic pressure difference.

(ii) Convective Mass Transfer - the rate of molecular diffusion of mass can be accelerated by the bulk motion of the fluid. Mass can be transported between the boundary of a surface and a moving fluid (drying of clothes, molecular diffusion of a sugar cube in a cup of coffee by stirring, moist air flowing over the surface of an ocean and precipitation on a dry land etc.), or between two moving fluids which are relatively immiscible (formation of clouds, vapourisation of water in a tea kettle). This mechanism of mass transfer is called 'convectIve mass transfer' and is analogous to heat transfer by convection (free or forced).

Dalton's Law of Partial Pressure

Each constituent of a multicomponent system contributes to the total pressure by an amount which is known as the 'partial pressure' of the constituent. The relationship between the partial pressures of the constituents is expressed by Dalton's Law:

The pressure of a mixture of gases is equal to the sum of the partial pressure of the constituents. The partial pressure of each constituent is that pressure which the gas would exert if it occupied alone that volume occupied by the mixture at the same temperature.

For a mixture of ideal gases, we have

$P = P_A + P_B + ... + P_K;$

where P_A is the partial pressure of the species A and so on.

$$= \sum_i P_i \qquad (1)$$

Dalton's law was reformulated by Gibbs to include a second statement on the properties of mixtures. The combined statement is Gibbs-Dalton law:

The internal energy. enthalpy and entropy of a gaseous mixture are respectively equal to the sum of the internal energies, enthalpies, and entropies of the constituents. The internal energy, enthalpy and entropy which a constituent would have if it occupied alone that volume occupied by the mixture at the temperature of the mixture.

Molar Density, Mass Density, Mass Fraction and Mole Fraction

There are a number of ways by which the concentration for a species in a multicomponent mixture can be defined:

(i) Molar Density or Molar Concentration, C_A = number of moles of the species A per unit volume of mixture, kg-mol/m^3

(ii) Mass Density or Mass Concentration, $\Box_A$ = mass of the species A per unit volume of the mixture, kg/m^3.

(iii) Mass Fraction, m_A = mass concentration of component A / total mass density of the mixture.

(iv) Mole Fraction, X_A = number of moles of species A / total number of moles of the mixture. = C_A/C

Therefore, the following summation rules hold true:

$C_A + C_B + ... + C_K = C$

$\Box_A + \Box_B + ... + \Box_K = \Box$

$X_A + X_B + ... + X_K = I1$

$rn_A + m_B + ... + m_K = I$ (2)

Since the number of moles = mass of species/molecular weight, we have

$$C_A = \rho_A / M_A$$

For a perfect gas, we have:

$P_A V = n_A R_o T$, where Ro is the universal gas constant,

and, $C_A = n_A / V = P_A / R_o T$

$X_A = C_A / C = P_A / P$ and $C = p / R_o T$

Mass Average and Molar Average Velocities and Different Types of Fluxes

Velocities: In a multicomponent mixture, the bulk velocity of the mixture can be defined on the basis of mass average or molar average velocity. Let V_A be the velocity of the species A and ρ_A is the mass density of the species A, then the mass average velocity would be:

$$V = \frac{\rho_A V_A + \rho_B V_B + ...}{\rho_A + \rho_B} = \frac{\rho_A V_A + \rho_B V_B + ...}{\rho}$$

$$= m_A V_A + m_B V_B + ... \qquad (3)$$

Similarly, the molar average velocity would be:

$$U = \frac{C_A V_A + C_B V_B + ...}{C_A + C_B} = \frac{C_A V_A + C_B V_B + ...}{C} = X_A V_A + X_B V_B + ...$$

Since mass transfer requires the diffusion of a species with respect to a plane moving with an average velocity, diffusion will take place when the diffusion velocity is in excess of the average velocity. Thus

Mass diffusion velocity of the species A : $V_A – V$ (4)

Molar diffusion velocity of the species A : $V_A - U$ (5)

Fluxes: The mass flux of species A can be expressed relative to either a fixed observer or an observer moving with the bulk velocity. For a stationary observer, the absolute flux of any species A will be equal to the sum of the flux due to the molecular diffusion and that due to the bulk motion.

Thus, Absolute flux: $\rho_A V_A$ and, Diffusion flux: $\dot{m} / A$

Flux due to bulk motion: $\rho_A V$

$$\rho_A V_A = \dot{m}/A + \rho_A V, \quad \text{or} \quad \dot{m}/A = \rho_A \left(V_A - V\right) \tag{6}$$

Similarly, molar diffusion flux = $C_A(V_A - V)$ (7)

Fick's Law of Diffusion*

The fundamental equation (one-dimensional) of molecular diffusion is known as Fick's law. It has been derived from the kinetic theory of gases, and can be written for a binary mixture as

$$J_A = -D_{AB} \, (d\,C_A/dx) \tag{8}$$

where D_{AB} = diffusion coefficient of species A with respect to species B, J_A = molar flux in the X-direction relative to the molar average velocity,

dC_A/dx = Concentration gradient in X-direction.

Let us consider a two compartment tank as shown in Fig. 5.1. One compartment contains gas A and the other compartment contains gas B and both the compartments are initially at a uniform pressure and temperature throughout. When the partition between the compartments is removed, the two gases will diffuse through each other until equilibrium is established and the concentration of the gases is uniform throughout the tank.

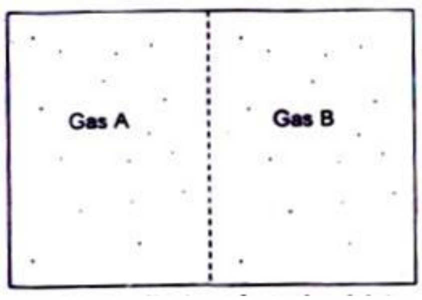

Fig. 1 Diffusion of species A in to species B

Fig 2 illustrates the dependence of diffusion on the concentration profile. The concentration of the species A on the left side of the imaginary plane is greater than that on the right side. As such, more molecules will cross the plane per unit time from left to right. This would lead to a net transfer of mass from the region of higher concentration to the region of lower concentration.

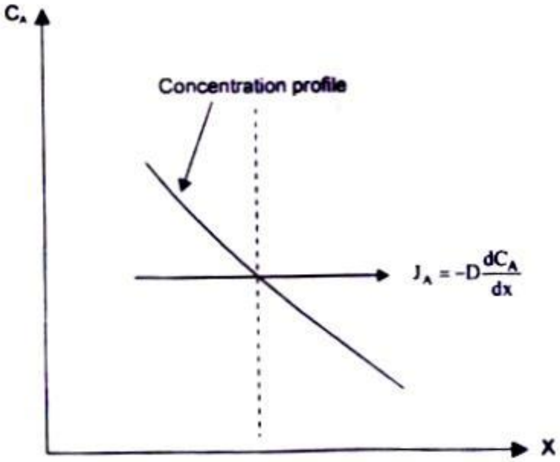

Fig.2 Dependence of diffusion on concentration profile

* This law assumes that fluxes are measured relative to the coordinates that move with the average velocity of the mixture.

Diffusion in Gases, Liquids and Solids

(i) Diffusion in Gases - the diffusion rates in gases are dependent on the molecular speed which is a function of temperature and therefore, the diffusion coefficient depends upon the temperature of gases.

Gilliland has proposed a semi-empirical equation for diffusion coefficient in a binary gas mixture –

$$D = 435.7 \frac{T^{3/2}}{p\left(V_A^{1/3} + V_B^{1/3}\right)^2} \left[\frac{1}{M_A} + \frac{1}{M_B}\right]^{1/2} \tag{9}$$

where D is in square centimeters per second, T is in Kelvin, p is the total pressure of the system in pascals, V_A and V_B are the molecular volumes of the species A and R as calculated from the atomic volumes in Table 12.1, M_A and M_B are the molecular weights of species A and B.

Diffusion coefficients for gases depend upon pressure, temperature and other molecular properties of diffusing gases. At two different pressure and temperature, we have

$$D_2 / D_1 = (p_1 / p_2).(T_2 / T_1)^{3/2} \qquad (10a)$$

Table 1 Atomic volumes*

Air	29.9	In secondary amines	1.2
Bromine	27.0	Oxygen, molecule (O_2)	7.4
Carbon	14.8	Coupled to two other elements:	
Carbon dioxide	34.0	In aldehydes and ketones	7.4
Chlorine		In methyl esters	9.1
Terminal as in R-Cl	21.6	In ethyl esters	9.9
Medial as in R-CHCl-R	24.6	In higher esters & ethers	11.0
Flourine	8.7	In acids	12.0
Hydrogen, molecule (H_2)	14.3	In union with S, P, N	8.3
in compounds	3.7	Phosphorous	27.0
Iodine	37.0	Sulphur	25.6
Nitrogen, molecule (N_2)	15.6	Water	18.8
in primary amines	10.5		

*(For three numbered ring like ethylene oxide, deduct 6.0, for four numbered ring like cyclobutane, deduct 8.5, for six numbered ring like benzene, deduct 15.6, for napthelene ring, deduct 30.0.)

Example: Calculate the diffusion coefficient of CO_2 in air at 30°C and at 1 atm pressure.

Solution: the atomic volumes are:

CO_2: Volume 34; molecular weight 44

Air: Volume 29.9; molecular weight 28.9

From Eq. (12.9), we get

$$D = 435.7\frac{\left(273+30\right)^{1.5}}{\left(1.0135\times10^{5}\right)\left(34^{1/3}+29.9^{1/3}\right)^{2}}\left(1/44+1/28.9\right)^{1/2}$$

= 0.1 cm^2/s

Estimate the diffusivity of ethyl alcohol (C2 Hs OH) in air at 25°C

and 1 atm pressure.

Solution: Kopp's law of additive atomic volumes applies in gases where compounds are involved.

For ethyl alcohol, the volume would be: 2(14.8) + 6(3.7) + 7.4 = 59.2

From Eq. (9), we have for ethyl alcohol,

$$D = 435.7\frac{\left(273+30\right)^{1.5}}{\left(1.0135\times10^{5}\right)\left(59.2^{1/3}+29.9^{1/3}\right)^{2}}\left(1/46+1/28.9\right)^{1/2}$$

= 0.753 cm^2/s

Similarly, for methane CH_4, the volume would be: 14.8 + 4(3.7) = 29.6

Using Eq (9), for methane, 0 = 1.114 cm^2/s.

(ii) Diffusion in Liquids and Solids - Diffusion in liquids occurs at much slower rate than in gases. Since kinetic theory of liquids is not as much developed as that of gases, it is usually assumed as a first approximation that equations of the same general form are applicable to the diffusion of a solute in a solvel1t as to the diffusion in gases, i.e., Fick's law is assumed valid for liquids.

Diffusion coefficient for most of the common organic and inorganic materials in the usual solvents such as water, alcohol and benzene at room temperature lie m the range of 1.79×10^{-3} to 1.075×10^{-7} cm^2/s.

Diffusion in solids is much slower than in liquids. Diffusion of solids in solid has limited engineering applications but diffusion of fluids in solids have extensive applications.

Fick's law is sometimes used, with an empirically determined effective diffusivity which takes care of the structure of solid. A typical problem of liquid transfer in a solid, of interest, is drying of solids.

The Equivalence of Diffusion Coefficient

Fick's law (Eq.8) can also be expressed in terms of mass flux per unit area or mass concentration or in terms of molal concentrations and fluxes. For gases, the law may be expressed in terms of partial pressures by making use of the perfect gas equation of state:

□ = □RT

Since the characteristic gas constant of a gas is: $R_A = R_o/M_A$

We have $□_A = □_A M_A/R_oT$

and $m_A / A = -D_{AB}\left(M_A / R_oT\right)dp_A / dx$ for isothermal diffusion. (5.10b)

Similarly, the diffusion of the component B, for the system shown in Fig. 5.1, we can write

$$\frac{\dot{m}_B}{A} = D_{BA}\,\frac{M_B}{R_oT}\,\frac{dp_B}{dx} \qquad (11)$$

When we have equimolal counter diffusion, shown in Fig. 12.3 (a, b), the steady state molal diffusion rates of the species A and B, represented by N_A and N_B will be given by

$$N_A = \frac{\dot{m}_A}{M_A} = -D_{AB}\left(\frac{A}{R_oT}\right)\left(\frac{dp_A}{dx}\right) \qquad (12)$$

and $$N_B = \frac{\dot{m}_B}{M_B} = +D_{BA}\left(\frac{A}{R_oT}\right)\left(\frac{dp_B}{dx}\right) \qquad (13)$$

The total pressure of the system remains constant at steady state,

or, $p = p_A + p_B$; and $dp_A/dx + dp_B/dx = 0$

as $dp_A/dx = -dp_B/dx$

Since each molecule of A is replaced by a molecule of B, the molal diffusion rates must be equal. Thus: $N_A = -N_B$, and

$$-D_{AB}\left(\frac{A}{R_0T}\right)\left(\frac{dp_A}{dx}\right) = -D_{BA}\left(\frac{A}{R_0T}\right)\left(\frac{dp_A}{dx}\right) \tag{14}$$

or $\qquad D_{AB} = D_{BA} = D$

This fact is known as the equivalence of diffusion coefficients or diffusivities in binary mixtures, and is a property of the binary mixture.

By integrating Eq. (12.10), we can obtain the mass flux of the species A as;

$$\frac{\dot{m}_A}{A} = -\frac{DM_A}{R_0T}\times\left(p_{A_2} - p_{A_1}\right)/\Delta x \tag{15}$$

corresponding to the nomenclature used in Fig. 3 (a, b). Table 2 gives the values of the binary diffusion coefficients.

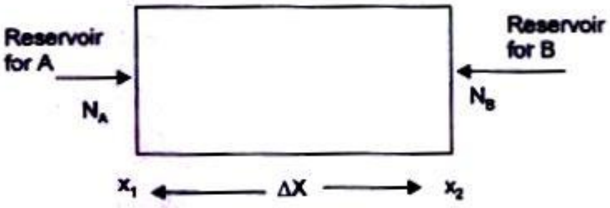

Fig. 12.3(a)

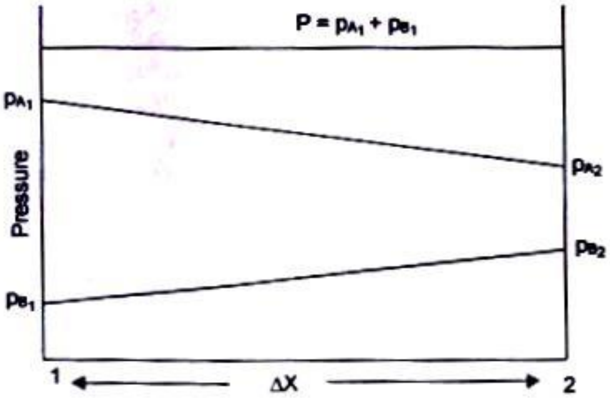

Fig. 3(b) Equlmolal counter-diffusion (partial pressure profile)

Table 2 Values of binary diffusion coefficient

Component A	Component B	T(K)	D_{AB}(m^2/s)
Solids			
O_2	Rubber	298	0.21×10^{-9}
CO_2	Rubber	298	0.11×10^{-9}
He	SiO_2	293	0.4×10^{-18}
Cd	Cu	293	0.27×10^{-18}
Al	Cu	293	0.13×10^{-33}
Dilute Solutions			
Caffeine	H_2O	298	0.63×10^{-9}
Ethanol	H_2O	298	0.12×10^{-8}
Glucose	H_2O	298	0.69×10^{-9}
Acetone	H_2O	298	0.20×10^{-8}
O_2	H_2O	298	0.24×10^{-8}
H_2	H_2O	298	0.63×10^{-8}
N_2	H_2O	298	0.26×10^{-8}
Gases			
NH_3	Air	298	0.28×10^{-4}
H_2O	Air	298	0.26×10^{-4}
CO_2	Air	298	0.16×10^{-4}
H_2	Air	298	0.41×10^{-4}
O_2	Air	298	0.21×10^{-4}
Acetone	Air	273	0.11×10^{-4}

Naphthalene	Air	300	0.62×10^{-5}
H_2	O_2	273	0.70×10^{-4}
H_2	N_2	273	0.68×10^{-4}
H_2	CO_2	273	0.55×10^{-4}
CO_2	N_2	293	0.16×10^{-4}
CO_2	O_2	273	0.14×10^{-4}
O_2	N_2	273	0.18×10^{-4}

Main Points of Fick's Law of Diffusion

These can be summarised as:

(i) It cannot be derived from first principles because it is based on experimental evidence.

(ii) It is valid for all phases of matter. Since mass transfer is strongly influenced by molecular spacing, diffusion is maximum in gases and minimum in solids.

(iii) It is analogous to Fourier law for conduction heat transfer.

(iv) It does not tell about diffusion due to temperature or pressure gradient or due to external forces.

Convective Mass Transfer Coefficient

When the mass is transported between the boundary of a surface and a moving fluid or between two moving fluids which are relatively immiscible, we have mass transfer by convection. The convective process can be either natural or forced, depending upon the existence of density or pressure gradient respectively, in the .medium. The convective mass transfer coefficient is defined in a manner similar to that for convective heat transfer.

Or, $$\dot{m}_A = h_{D_A} A\left(C_{A_1} - C_{A_2}\right) \tag{1}$$

where $\dot{m}_A$ = diffusive mass flux of species A, h_{D_A} = mass transfer coefficient, and

C_{A_i}, C_{A_2}, are the concentrations through which diffusion takes place. The convective mass transfer coefficient depends upon the fluid properties, the mechanism of fluid flow, and the geometry of the flow system and is analogous to the convective heat transfer coefficient.

For a steady-state diffusion across a layer of thickness L,

$$\dot{m}_A = DA\left(C_{A_1} - C_{A_2}\right)/L = h_{D_A} A\left(C_{A_1} - C_{A_2}\right)$$

therefore, $h_{D_A} = D/L$, m/s (2)

Dimensionless umbers Used in Mass Transfer

The following dimensionless numbers are of significance in convective mass transfer:

Schmidt Number, Sc = momentum diffusivity/mass diffusivity = □/D = □/□D.

This number is analogous to Prandtl number and when Sc = I, there would be complete similarity between momentum and concentration equations and the hydrodynamic results may be applied directly to convective mass transfer problems.

Sherwood number, Sh = $h_D x/D$, and IS analogous to Nusselt number. Lewis Number, Le = aID, the temperature and concentration profiles will be similar when the Lewis number is equal to unity.

Mass Grashof number, $Gr_m = \dfrac{gL^3\left(C_s - C_\infty\right)}{v^2 C_\infty}$

C_s = mass concentration at surface `

C_∞ = free stream mass concentration

An Expression for the Convective Mass Transfer Coefficient for Laminar Flow Over a Flat Plate

Let us consider a flat plate where the concentration at the surface of the species A is different than its concentration in the free stream. The species A will diffuse into the fluid and a concentration boundary layer will develop as shown in Fig.8. The thickness of the concentration boundary layer is defined ill the same manner as that of the hydrodynamic boundary layer or thermal boundary layer.

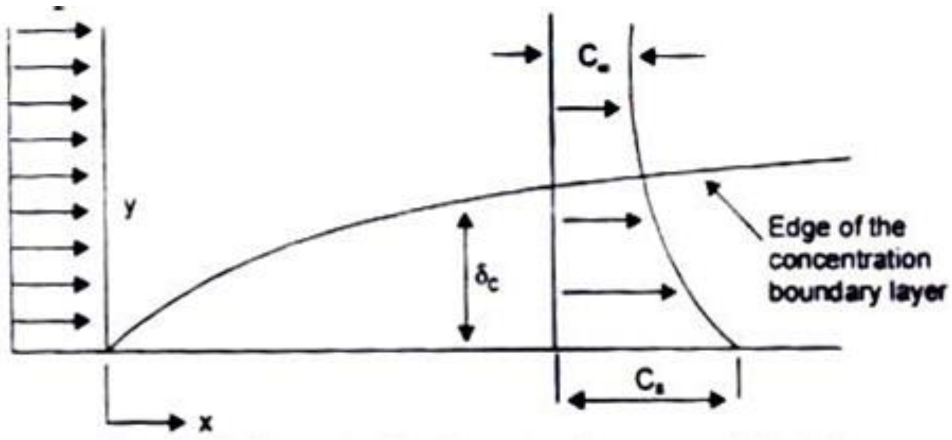

Fig. 8 Concentration boundary layer on a flat plate

Since there is a remarkable similarity between the laws governing the boundary layer growth of the three processes: momentum, heat and mass, the governing equation far the concentration boundary layer can be written as:

$$u\,.\,\partial C_A/\partial x+v.\partial C_A/\partial y=D.\partial^2 C_A/\partial y^2 \qquad (3)$$

And for laminar flow over the flat plate, the average values for the convective mass transfer coefficient can be obtained from the relation:

$$Sh_L=h_D L/D=0.664\ Re_L^{0.5}\,Sc^{1/3}\ \ (\text{for } Sc\geq 0.6) \qquad (4)$$

When the boundary layer is partly laminar and partly turbulent, transition occuring at Re = 5 × 10^5, the correlation for mass transfer would be, similar to heat transfer, and is given by

$$Sh_L=\left(0.037\,Re_L^{0.8}-870\right)Sc^{1/3} \qquad (5)$$

For flow over smooth flat plates, the Colburn analogy predicts:

(a) Laminar flow: $C_f/2=0.664\,Re_L^{-0.5}=h_D/U_\infty Sc^{2/3}$

(b) Turbulent flow: $C_f/2=\left(h_D/U_\infty\right)Sc^{2/3}=0.0296\ Re_L^{-0.2}$ (6)

Expressions for Convective Mass Transfer Coefficient for Flow through Tubes, Flow over Spheres and Cylinders

We have forced convection mass transfer when a liquid evaporates from the wetted walls of a tube into the gas flowing through that tube. A concentration boundary layer develops inside the tube, similar to the hydrodynamic boundary layer, as shown in Fig. 12.9.

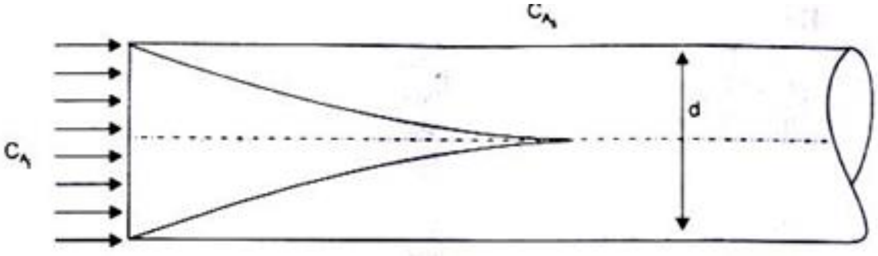

Fig. 9 Concentration profile for mass transfer in a tube

For fully developed velocity and concentration profiles in a laminar flow through a tube, the following equations have been suggested for the evaluation of mass transfer coefficients:

$h_D d/D = Sh = 3.66$ for uniform wall mass concentration (7)

$Sh = 4.34$ for uniform wall mass flux (8)

In turbulent flow, the concentration of the diffusing component varies with time and space and as such mass transfer coefficients are evaluated either experimentally or with the help of empirical equations. These equations are based on analogy with heat transfer.

Gilliland has proposed the following equation for the vapourization of liquids from the walls of smooth circular tubes when air is forced to flow through the tube: $Sh = 0.023(Re)^{0.83} (v/D)^{0.44}$; $2000 < Re < 35{,}000$;

and for gases $0.6 < Sc < 2.5$ (9)

The Reynolds and Colburn analogy can also be used to calculate the mass transfer coefficient from the friction factor. Or,

$$\frac{h_D}{U_\infty} . Sc^{2/3} = f/8 = \frac{C_f}{2} \qquad (10)$$

When the coefficient of friction C_f is eliminated from Colburn analogy for heat and mass transfer, we get

$$\mathrm{St}(\mathrm{Pr})^{2/3} = \mathrm{St_m}(\mathrm{Sc})^{2/3}$$

$$\text{or,}\quad \frac{h}{\rho C_p U_\infty}(\mathrm{Pr})^{2/3} = \mathrm{St_m}(\mathrm{Sc})^{2/3}$$

$$\therefore\ \frac{h}{h_D} = \rho C_p \left(\frac{\mathrm{Sc}}{\mathrm{Pr}}\right)^{2/3} = \rho C_p \left(\frac{\alpha}{D}\right)^{2/3} = \rho C_p (\mathrm{Le})^{2/3} \qquad (11)$$

Eq. (11) gives a relation between the mass transfer and heat transfer coefficients. That is, the coefficient of mass transfer can be evaluated from the data available for heat transfer coefficients. Eq. (11) is known as Lewis Equation. Lewis Equation $\mathrm{Le} \approx 1$ in gas-vapour mixture.

Analysis among Mass, Heat and Momentum Transfer

Analogies among mass, heat and momentum transfer have their origin either in the mathematical description of the effects or in the physical parameters used for quantitative description.

To explore those analogies, it could be understood that the diffusion of mass and conduction of heat obey very similar equations. In particular, diffusion in one dimension is described by the Fick's Law as

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

Similarly, heat conduction is described by Fourier's law as

$$q = -k\frac{dT}{dz}$$

Where k is the thermal conductivity.

The similar equation describing momentum transfer as given by Newton's law is

$$\tau = -\mu \frac{dv}{dz}$$

Where τ is the momentum flux (or shear stress) and μ is the viscosity of fluid.

At this point it has become conventional to draw an analogy among mass, heat and momentum transfer. Each process uses a simple law combined with a mass or energy or momentum balance.

In this section, we shall consider several analogies among transfer phenomenon which has been proposed because of the similarity in their mechanisms. The analogies are useful in understanding the transfer phenomena and as a satisfactory means for predicting behaviour of systems for which limited quantitative data are available.

The similarity among the transfer phenomena and accordingly the existence of the analogies require that the following five conditions exist within the system

1. The physical properties are constant
2. There is no mass or energy produced within the system. This implies that there is no chemical reaction within the system
3. There is no emission or absorption of radiant energy.
4. There is no viscous dissipation of energy.
5. The velocity profile is not affected by the mass transfer. This implies there should be a low rate of mass transfer.

Reynolds Analogy

The first recognition of the analogous behaviour of mass, heat and momentum transfer was reported by Osborne Reynolds in 1874. Although his analogy is limited in application, it served as the base for seeking better analogies.

Reynolds postulated that the mechanisms for transfer of momentum, energy and mass are identical. Accordingly,

$$\frac{k_c}{v_\infty} = \frac{h}{\rho v_\infty C_p} = \frac{f}{2}$$

Here h is heat transfer coefficient

f is friction factor

ν_∞ is velocity of free stream

The Reynolds analogy is interesting because it suggests a very simple relation between different transport phenomena. This relation is found to be accurate when Prandtl and Schmidt numbers are equal to one. This is applicable for mass transfer by means of turbulent eddies in gases. In this situation, we can estimate mass transfer coefficients from heat transfer coefficients or from friction factors.

Chilton – Colburn Analogy

Because the Reynold's analogy was practically useful, many authors tried to extend it to liquids. Chilton and Colburn, using experimental data, sought modifications to the Reynold's analogy that would not have the restrictions that Prandtl and Schmidt numbers must be equal to one. They defined for the j factor for mass transfer as

$$j_D = \frac{k_c}{v_\infty}(Sc)^{2/3}$$

The analogous j factor for heat transfer is

$$j_H = St \ \mathrm{Pr}^{2/3}$$

where St is Stanton number = $\dfrac{Nu}{\mathrm{Re}\ \mathrm{Pr}} = \dfrac{h}{\rho \vartheta_\infty C_p}$

Based on data collected in both laminar and turbulent flow regimes, they found

$$j_D = j_H = \frac{f}{2} \quad \text{----------------------------- (4.38)}$$

This analogy is valid for gases and liquids within the range of $0.6 < Sc < 2500$ and $0.6 < \mathrm{Pr} < 100$.

The Chilton-Colburn analogy has been observed to hold for many different geometries for example, flow over flat plates, flow in pipes, and flow around cylinders.

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SCHOOL OF BIO AND CHEMICAL ENGINEERING

DEPARTMENT OF CHEMICAL ENGINEERING

HEAT AND MASS TRANSFER FOR BIOLOGICAL SYSTEMS

SCHA1311

UNIT – 2 BASIC SEPARATION PROCESSES-1- SCHA1311

UNIT 2

BASIC SEPARATION PROCESSES

Introduction to separation processes, Distillation, Types of distillation – Simple, Steam, Vacuum, Continuous distillation,absorption-packed and plate columns, Adsorption-chemisorption, physical adsorption, isotherms, membrane separation process – Electrodialysis, Reverse osmosis, Ion exchange, Extraction – Batch and Continuous operations.

INTRODUCTION

The separation processes are divided in equilibrium and rate governed separation process. In equilibrium governed separation processes, the product phases are in equilibrium with the inlet phases. In rate governed separation processes, difference of rate of physical transport of species brings the separation. Some of the specific separation processes under these two categories are elaborated below.

EQUILIBRIUM GOVERNED PROCESS

Four popular and quite common equilibrium governed separation processes are described.

Distillation

Distillation is based on the differences in boiling points of the constituents in a mixture. The component having the lower boiling point will go to the vapor phase earlier, leaving behind the other component in the residual mixture. In this process, external heating is required to heat up the system. Therefore, the top vapor phase is condensed and is rich in one component and the residual liquid phase is rich in other component. This is known as flash distillation. In another process, a part of the condensed stream from the top is recycled down the column. This is known as reflux. In this case, there is intimate mixing of the liquid stream coming down and vapor going up. This operation is carried out in counter current fashion and this enhances the efficiency of the process. This is generally used for separation of aqueous mixture of various organic solvents, like, toluene, benzene, acetone, ethyl benzene, etc.

Absorption

In this process, a vapor in an inert gas is absorbed in a liquid stream. The vapor is soluble in the liquid. The liquid stream rich in solubilized vapor components is separated by distillation or some other suitable techniques. The liquid can be recovered and reused in the process itself. An example is removal of ammonia from ammonia – water mixture using water as solvent. Removal of carbon di oxide from a mixture of air and carbon dioxide using a solvent like primary or secondary or tertiary amines is another example. Typically this operation is carried out in counter current manner to enhance the efficiency of this process. Sometimes, the packed beds or staged beds like trays are used to have better mixing during the transfer process. It may be mentioned that absorption is a bulk phenomena, i.e., the gaseous solutes are transported to the bulk of the liquid stream. It may be noted that to effect separation, a matter is introduced in the system.

Adsorption

Separation of solutes by transporting it from gaseous or liquid streams on a solid surface is known as adsorption. As opposed to absorption it is a surface phenomenon. The solute is fixed on the solid surface only. Typically, the solid surface is specific for a particular component present in the feed stream. For example, in a liquid stream having organic as well as inorganic components, the organics are preferentially adsorbed on the surface of activated carbon. Typically, in a bed, the adsorbent particles are kept and the fluid is pumped through the system for intimate mixing. In the process, the solute is transferred to the solid phase. After sometime, the solid phase is completely saturated and the transfer of species becomes extremely slow due to lack of driving force. In such case, the bed has to be replaced by new adsorbents or the bed has to be regenerated by a suitable washing protocol. It may be noted, that a reasonably lower particle size of adsorbents provides sufficient surface area for separation. Removal of organics from an aqueous mixture using activated carbon is an example of adsorption. It may be noted that to effect separation, a matter is introduced in the system.

Drying

In drying, water vapors from a solid material are removed by using a vapor stream to an acceptable small limit. Generally it is carried out by applying thermal energy as compared to introduction of a matter in case of absorption or adsorption. The process efficiency depends on the temperature, relative humidity and other thermo physical properties of the drying vapor as well as the temperature and other thermo-physical properties of the solid.

RATE GOVERNED PROCESS

Most of the membrane based processes are the rate governed. The main crux of the separation lies in the difference of transport of various species through the membrane. The driving force of such transport is generally gradient of chemical potential. If we recall the definition of gradient of chemical potential, it is composed of four parts. These are concentration gradient, pressure gradient, temperature gradient and electrochemical potential gradient. Presence of these causes (one or more of such gradients) results into effects (the difference in separation and hence effects). Some of these systems are described below.

Osmosis

Osmosis is observed when a solution is separated from the solvent by a semipermeable (only solvent is permeable species) membrane. Apparently, difference in osmotic pressure across the membrane causes the transport of the solvent. Actually, osmotic pressure is a colligative property and it is a function of concentration. Therefore, the concentration (or more generally activity) gradient of the solvent is the main driving force in osmosis. In this case, the solvent flows from the solvent side to the solution side until, the solvent activity becomes almost equal in both the sides. This results in generation of hydrostatic pressure difference between the two sides, known as osmotic pressure. If the initial concentration of solute is more in the solution side, more solvent will flow in from the solvent side to equalize the solvent activity on both the sides, resulting in more osmotic pressure difference.

Reverse Osmosis

It must be understood that osmosis is a natural phenomenon. In case of reverse osmosis, pressure is applied on the solution side externally (using a pump or a compressor), so that the osmotic pressure is overcome and the solvents are forced out of the solution side. The efficiency of this process depends on the porosity, morphology and thickness of the membrane. These factors influence the transport of the solvent through the membrane immensely and thereby, dictating the throughput of the process and the quality of the product. Since, reverse osmosis membranes have very small pore size, this is used for separation of lower molecular weight species, like, salts. A salt having very osmotic pressure, the operating pressure in the feed side of a reverse osmosis process is therefore extremely high. This will lead to deposition of salts near the membrane surface, leading to build up of a concentration boundary layer. Therefore, the pressure gradient in this system leads to concentration difference.

Dialysis

In this case, two liquid streams are separated by a permeable barrier or membrane. In the feed side, a specific set of solutes are permeated through the membrane to the other side. The upstream feed is known as the feed side and the downstream is known as the dialysate. Typically, dialysate stream is pure distilled water. Thus, the concentration gradient between the two streams is the maximum. The transport is effected by the concentration gradient between two streams. The duration of separation entirely depends on the rate

DISTILLATION

DEFINITION

Distillation may be defined as the separation of the constituents of a mixture including a liquid by partial vaporization of the mixture and separate and collect the vapor.

Such separation may include

(i) one liquid from non-volatile impurities.
(ii) one liquid from one or more other liquids, with which it may be miscible, partially-miscible or immiscible

N.B.

In practice it is difficult to distinguish between evaporation, distillation and drying.

Based on the intention:

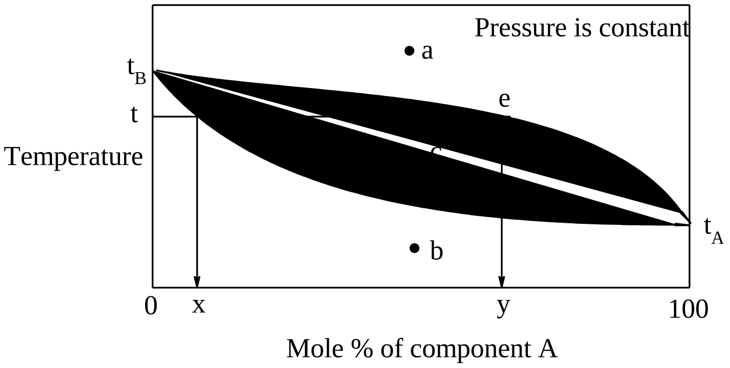
(i) when condensation vapor is required the operation is called distillation
(ii) when the concentrated liquid residue is required the operation is called evaporation.
(iii) when the dried solid residue is required as product the process is called drying

BOILING POINT DIAGRAM OF A BINARY MIXTURE

The figure represents the boiling point and equilibrium-composition relationship, at constant pressure.

Two liquids A (b.p. t_A) and B (b.p. t_B) are taken in a chamber of constant pressure. Now at any temperature the vapor composition and liquid composition will give two lines when plotted vs. temperature.

In boiling point diagram, temperatures are plotted as ordinates and compositions as abcissas.



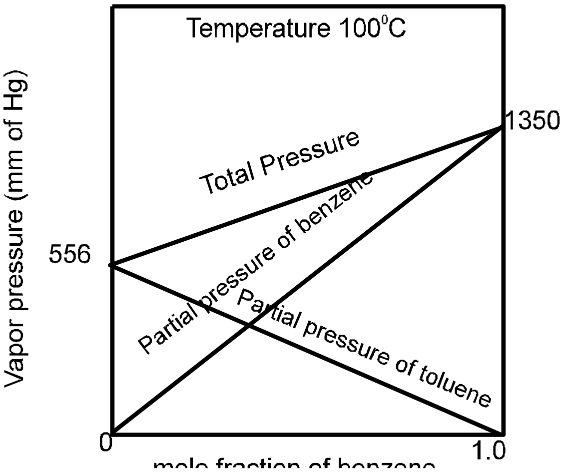
- The diagram consists of two curves, the ends of which coincide with the b.p. of two components (t_A and t_B).
- The upper-curve describes vapor composition and lower-curve liquid composition.

- At any temperature t the horizontal line cuts the vapor composition curve at ‘e’ which corresponds to vapor composition of y (mole%A) and cuts the liquid composition curve at ‘d’ which corresponds to liquid composition of x (mole% of A). So any two points on the same horizontal line (such as d and e) represent compositions of liquid and vapor in equilibrium at temperature ‘t’.
- For all points above the top line (such as point ‘a’) the mixture is entirely vapor.
- For all points below the bottom line (such as point ‘b’) the mixture is completely liquefied.
- For all points between the two curves (such as point ‘c’) the system consists partly of liquid and partly of vapor.

RAOULT’S LAW

Raoult’s law states that, any particular temperature, the partial pressure of one component of a binary mixture is equal to the mole fraction of that component multiplied by its vapor pressure in the pure state at this temperature.

e.g. to illustrate Raoult’s law, let us consider the case of benzene and toluene mixture.



At a temperature of 100^0C toluene has a vapor pressure of 556 mm. Consequently, if partial pressure is plotted against composition, the partial pressures of toluene at various compositions will fall along a straight line from 556 mm for pure toluene to zero for pure benzene. At this same temperature benzene has vapor pressure of 1350 mm, and its vapor pressure will change linearly from zero for 0% benzene to 1350 mm for pure benzene.

The total pressure for any composition will be the sum of the two partial pressures at that composition.

If the partial pressures are straight lines i.e. Raoult’s law holds then the total pressure will be a straight line between 556 m for pure toluene and 1350 mm for pure benzene.

Derivation

Two liquids A and B are at constant temperature.

Liquid A is more volatile than B.

If Raoult’s law holds for this binary mixture then

from Raoult’s law $p_A = P_A x$ (1)

and $p_B = P_B (1 - x)$

where p_A and p_B = partial pressure of A and B respectively

P_A and P_B = vapor pressure of pure A and B

x = mole fraction of A in the solution

If P represents the total pressure, then

$$P = p_A + p_B \qquad (2)$$

$$= P_A x + P_B(1 - x)$$

From Dalton's law

$$y = \frac{p_A}{p_A + p_B}$$ where y = mole fraction of A in vapor phase

$$= \frac{P_A x}{P}$$ [from (1) and (2)]

RELATIVE VOLATILITY

For a more volatile phase in equilibrium with a liquid phase, the relative volatility of component A (the more volatile component) with respect to component B is defined by the equation:

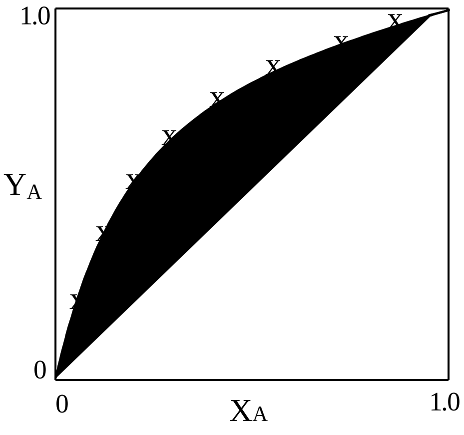
$$\alpha_{AB} = \frac{y_A / x_A}{y_B / x_B}$$

where

α_{AB} = relative volatility of component A with respect to component B

y = mole fraction of component A in vapor phase

x = mole fraction of component in liquid phase



Equilibrium curve

In case of binary system, $y_B = 1 - y_A$ and $x_B = 1 - x_A$.

Substituting,

$$\alpha_{AB} = \left(\frac{y_A}{1 - y_A}\right)\left(\frac{1 - x_A}{x_A}\right)$$

Rearranging we get $Y_A = \dfrac{\alpha_{AB} X_A}{1 + (\alpha_{AB} - 1) X_A}$

Equilibrium curve

If α_{AB} is given then from the above equation a set of X_A and Y_A can be calculated. When Y_A is plotted against X_A the curve is called *equilibrium curve*.

If the liquid phase obeys Raoult's law and the vapor phase obeys Dalton's law then,

$$y_A = \frac{P_A x_A}{P}$$

So $\alpha_{AB} = \dfrac{P_A x_A / P}{P_B (1 - x_A) / P} \; \dfrac{1 - x_A}{x_A} = \dfrac{P_A}{P_B}$

HENRY'S LAW

The partial pressure of a component over a solution is proportional to its mole fraction in the liquid . This can be expressed as $p_A = C x$

where p_A = partial pressure of component A

x = mole fraction of A in liquid phase

C = Henry's law constant; C is constant only at constant temperature

N.B. Raoult's law is essentially a special case of Henry's law where the constant C in equation is the vapor pressure of the pure component.

DISTILLATION METHODS

A. Distillation methods for miscible liquid systems

1. Equilibrium or Flash Distillation
2. Simple or Differential Distillation
3. Fractional Distillation
4. Distillation under reduced pressure (e.g. Molecular Distillation)
5. Special Distillation Methods for non-ideal mixtures
    (a) Distillation of Azeotropic Mixtures
    (b) Extractive Distillation

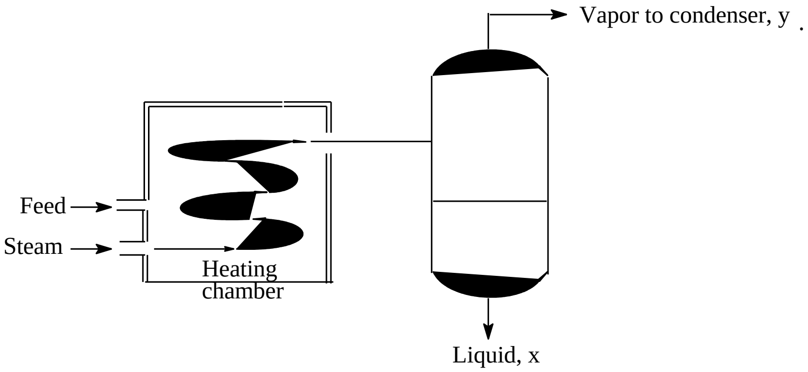
(B) Distillation of immiscible liquids (e.g. Steam Distillation)

1. EQUILIBRIUM DISTILLATION / FLASH DISTILLATION

There are two types of distillations that do not involve rectification

(a) Equilibrium distillation or flash distillation and

(b) Simple or differential distillation.



(a) Equilibrium distillation or flash distillation

This is a single stage operation where a liquid is partially vaporized, the vapors are allowed to come in equilibrium with the residual liquid and the resulting vapors and liquid are separated.

Use: This method is used only when the difference between volatilities of two components is very large

Let us consider a binary system whose components are A and B. A is more volatile.

- Feed: W_F = number of moles of liquid fed

  x_F = mole fraction of component A in feed

- Suppose V moles are vaporized in an equilibrium-distillation process.

Now in

Liquid phase

Number of moles left in liquid phase = $(W_F - V)$ moles

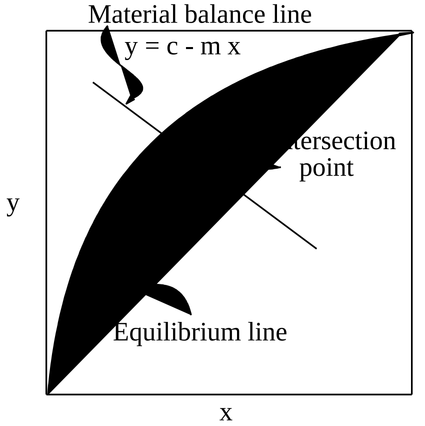
Let the composition of the residual liquid = x mole fraction of A

Vapor phase

Composition of vapor phase = y mole fraction of A

Number of moles gained = V

Form material balance equation with respect to A



Equilibrium curve of A-B system

Moles of A at start = Moles of A in vapor phase

+ Moles of A in liquid phase

$$W_F x_F = V y + (W_F - V) x$$
(i)

In this case all the parameters are known except x and y .

2nd equation required for solving is obtained from equilibrium curve of the A, B system.

Eqn(i) is a straight line,

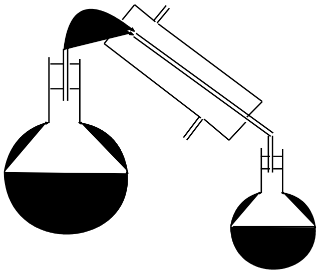
$$V y = W_F x_F - (W_F - V) x$$

or, $$y = \left(\frac{W_F x_F}{V}\right) - \left(\frac{W_F - V}{V}\right) x$$

or, $$y = c - m x$$

Plotting this equation in the equilibrium curve the point of intersection is obtained. The value of x and y can be obtained form the point of intersection.

2. SIMPLE / DIFFERENTIAL DISTILLATION



In this process vapor is removed from the system as soon as it is formed and condensed.

Use:

- This method is commonly used in laboratory
- In industries it is only used for systems having high relative volatilities.

Derivation of Raleigh's equation

Let us consider a batch of W_0 moles of liquid was taken at the beginning.

Suppose at any given time during distillation there are W moles of liquid left in the still.

At this time let the mole fraction of A in liquid is *x*.

Suppose a very small amount of liquid dW is vaporized. In the vapor phase the mole fraction of component A is y.

	At a given time	After a moment
Total moles of liquid present	W	W – dW
Moles of A present in liquid	Wx	(W – dW)(x – dx)
Total moles of liquid removed		dW
Moles of A present in the vapor		y

Therefore a material balance equation with respect to A will be

$$xW = (W - dW)(x - dx) + y\,dW$$

or, $\cancel{xW} = \cancel{xW} - x\,dW - W\,dx + dW\,dx + y\,dW$

dW dx is very small hence ignoring the term the equation will be

$$y\,dW - x\,dW = W\,dx$$

or, $$(y - x)\,dW = W\,dx$$

or, $$\frac{dW}{W} = \frac{dx}{y - x}$$

Now, integrating between the limits

$$\int_{W_0}^{W_1} \frac{dW}{W} = \int_{X_0}^{X_1} \frac{dx}{y - x} \qquad \text{or,} \qquad \boxed{\ln\frac{W_1}{W_0} = \int_{X_0}^{X_1} \frac{dx}{y - x}}$$

This equation is known as Raleigh's equation. It relates the amount of material distilled with instantaneous composition of the liquid at that moment

The function $\frac{dx}{y - x}$ can be integrated graphically from the equilibrium curve, since the curves gives the relationship between x and y.

N.B.

x	y	y – x	1/(y – x)
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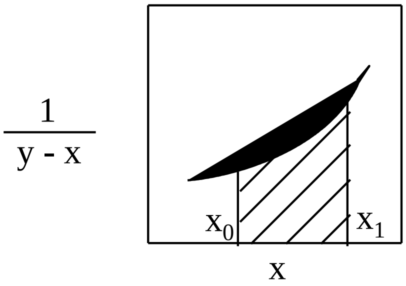


Application of Raleigh's Equation

1. By using the Raleigh's equation the effectiveness of simple distillation for a given system can be estimated. [*Ref: A.B.Paradkar, Introduction to Pharmaceutical Engineering, p. 247*]
2. It is used in determination of cut-off point when we can stop distillation as soon as the vapor composition falls below the required purity of the product.

SIMPLE DISTILLATION

Objective

Simple distillation is the process of converting a liquid into its vapors which, are passed through a cooling surface to condense the vapors. The condensed vapors are reformed into liquid which, is collected in a receiver.

Apparatus for laboratory scale

It consists of a distillation flask with a side arm sloping downward that is connected to a condenser. The condensed vapors are collected in a flask called '*receiver'*. The whole apparatus is made of glass.

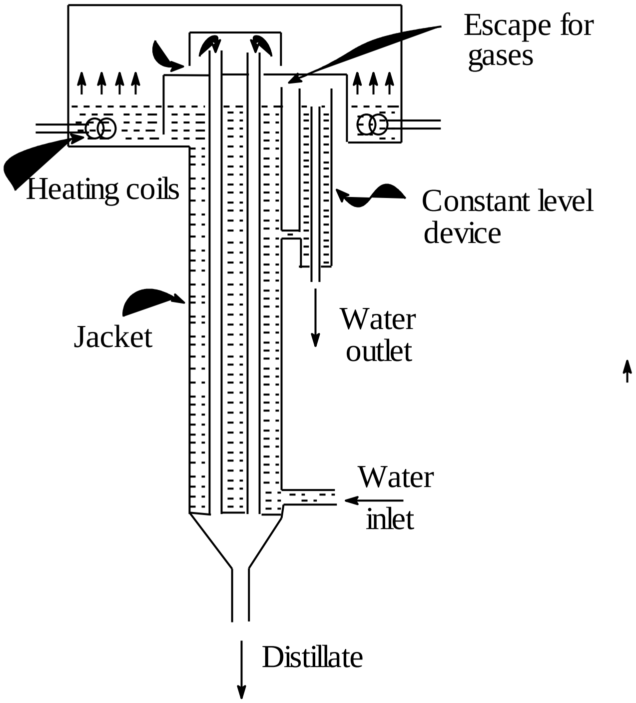


Fig. Distillation unit for purified water

A thermometer is fitted in the distillation flask to note down the temperature at which, the vapors are distilled.

Bumping is avoided by adding small pieces of porcelain or porous pot before distillation.

Apparatus for preparation of purified water

The boiler may be made of cast iron but the baffles and the condenser tubes that comes into contact with product are made of stainless steel or monel metal.

The cold water from the water tap enters the still through the inlet, which rises in the jacket fitted with a constant level device, the excess of water over flow through the outlet.

A portion of hot water at 90 to 95^0C enters into the boiler through a narrow opening – the level of water is maintained in the boiler up to overflow level.

The water is boiled in the boiler by means of heating coils. On heating, the dissolved gases in the condenser are allowed to escape through a small opening and only the steam escapes into the condensing tubes.

Since the dissolved gases are more volatile than water they escape in the first portion of the distillate, therefore, must be rejected. Similarly, the last portion may contain volatile portion of the dissolved solid substances in tap water – hence, discarded.

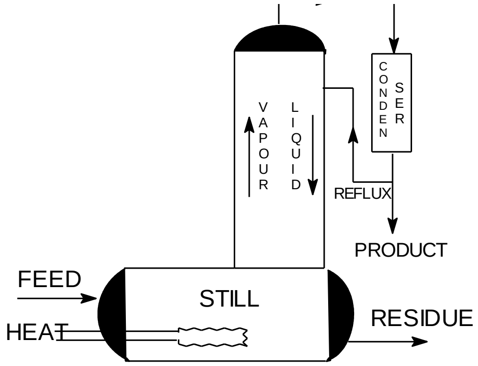
Application of simple distillation in pharmacy

1. It is used for the preparation of distilled water and water for injection.
2. Many volatile oils and aromatic waters are prepared by simple distillation e.g. Spirit of nitrous ether and Aromatic Spirit of Ammonia
3. Concentration of liquid and to separate non-volatile solid from volatile liquids such as alcohol and ether.

3. FRACTIONAL DISTILLATION / RECTIFICATION

A rectifying unit consists primarily of

(a) a still or reboiler, in which vapor is generated,
(b) a rectifying or fractionating column through which this vapor rises in counter-current contact with a descending stream of liquid, and
(c) a condenser, which condenses all the vapor leaving the top of the column, sending part of this condensed liquid (the reflux) back to the column to *descend* counter to the rising vapors, and delivering the rest of the condensed liquid as product.



As the liquid stream descends the column, it is progressively enriched with the less volatile constituent.

The top of the column is cooler than the bottom, so that the liquid stream becomes progressively hotter as it descends and the vapor stream becomes progressively cooler as it rises. This heat transfer is accomplished by actual contact of liquid and vapor, and for this purpose effective contact is desirable.

CONSTRUCTION OF RECTIFYING COLUMN

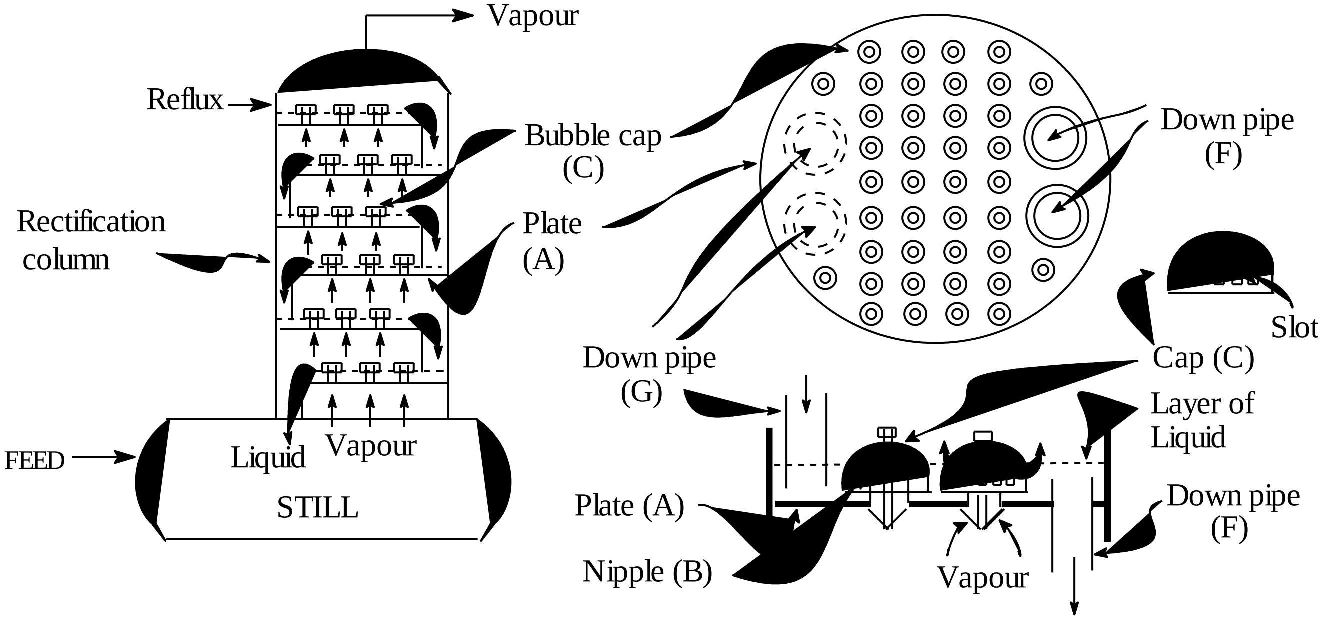
There are different varieties of equipments for rectification

(a) Plate column (i) Bubble cap column

(ii) Sieve-plate column

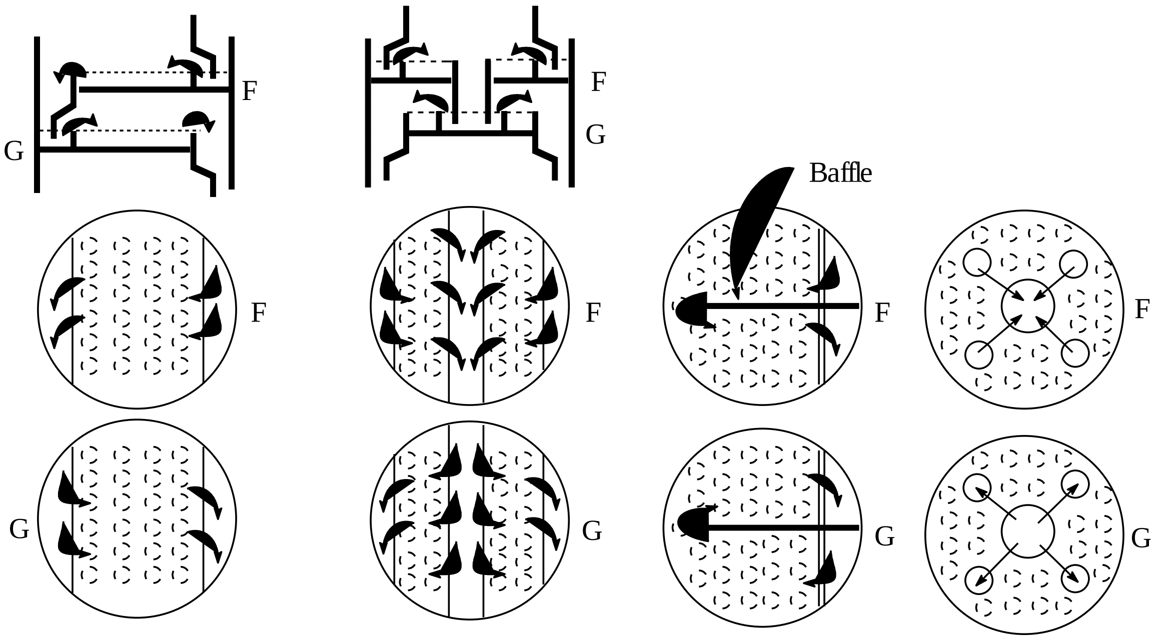
(b) Packed column

BUBBLE-CAP COLUMN



- The column is divided into sections by means of a series of *horizontal plates* A.
- Each plate carries a number of short *nipples* B (or *riser*). Each nipple is covered by a bell-shaped *cap* C that is secured by a spider and bolt with the plate. The edge of the cap is *serrated* or the sides may be *slotted*.
- Vapor rises from the plate below through the nipple, is diverted downward by the cap, and bubbles out under the serration or through the slots.
- A layer of liquid is maintained on the plate by means of an *overflow* or *down-pipe* (F) and the depth of the liquid is such that the slots are submerged.
- The *down-pipe,* (G) from the plate above, is sealed by the liquid on the plate below, so that the vapor cannot enter the down-pipe.
- Ordinarily, the liquid is delivered at one end of a diameter by the down-pipe from the plate above, flows the other end of the same diameter.

Types of down-comers



Cross flow Split flow Reverse Flow Radial Flow

(a) Cross flow

The liquid flows across the plate from right to left on plate F and left to right on plate H and so on down the column.

(b) Split flow

On plate F the liquid flows form the two sides to the center. On plate H it flows from the center to the two sides and so on down the column. This arrangement is commonly known as split flow.

(c) Reverse flow

Liquid comes down the space on one side of the baffle and flows across the plate from right to left, around the end of the baffle, from left to right and down the space behind the weir. This arrangement is called reverse flow.

(d) Radial flow with circular down-take

One plate will have four or more down-comers around the circumference, and the next plate will have a down-comer at the center so that on the upper plate the flow is from the circumference towards center and on the next plate the flow is from the central down-take to the circumference.

Specification of bubble cap rectification column

Column diameter	2 to 15 ft	
Height	few feet to over 100 ft	
Bubble cap diameter	3 to 6 inches	
Slots in a 3 inches bubble cap may be	1/8 to 3/32	inch wide
	½ to 1	inch height

SIEVE PALTE COLUMNS

All the constructions are same as bubble cap columns. Instead of bubble cap plates, flat plates with a large number of relatively small perforations, drilled in them are used. These perforations are usually 3/16 to ¼ inch in diameter.

The velocity of the vapor through these holes is sufficient to produce the liquid running down the holes.

PACKED COLUMNS

The column is entirely filled with some sorts of material that offers a large surface area supposedly wetted by the liquid.

A large variety of materials are used among which **Raschig rings** are popular. A Raschig ring is a hollow cylinder whose length is equal to its diameter. This may be made of metal (by sawing sections off a pipe), stone ware, ceramics, carbon, plastics, or other materials. Raschig rings are usually dumped at random in the column.

Raschig Ring Lessing Ring Pall Ring Berl Saddle Intalox Saddle

Advantages

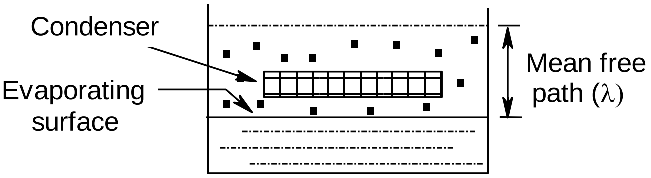
(i) Have a low pressure drop per unit of height than bubble cap
(ii) For very small diameters of column, where it would be difficult to get in more than two or three bubble caps, a packed column can be used.
(iii) Since Raschig rings can be made of any material, hence packed columns can be used for corrosive materials.
(iv) The amount of liquid held up in the column is low so thermolabile liquid remains in contact with high temperature for a short time than bubble cap method.

Disadvantages

(i) They are relatively inflexible.
(ii) Distribution of liquid uniformly in such packed column is difficult. It is found that, as the liquid passes down the tower it tends to concentrate at the walls and leave the center dry.

4. DISTILLATION UNDER REDUCED PRESSURE / VACUUM DISTILLATION

Theroy



Liquid boils when its vapor pressure is equal to the atmospheric pressure. Liquids, which are decomposed at their boiling point under atmospheric pressure, can be distilled at a much lower temperature than its boiling point if the pressure is reduced on the surface of the liquid. Boiling under reduced pressure will also increase the rate of distillation.

Molecular Distillation

Theory / Principle of Molecular Distillation

In a high vacuum distillation operation, where the material distills from an evaporating surface to a relatively cool condensing-surface. The conditions are such that, the mean free path of the distillating molecules is greater than the distance between the evaporating and condensing surface.

- The vacuum applied in these types of apparatus is about 1 μm Hg pressure or less.

<u>*Mean free path*</u> *is defined as the average distance traveled by the molecules in a straight line without any collision.* It can be calculated by Clausius law:

$$\lambda = \frac{1}{\sqrt{2\pi d^2 N}}$$

where, λ = mean free path (cm)

d = diameter of the molecules (cm)

N = number of molecules in 1 cm^3 volume.

It is clear from the above equation and chart that, mean free path can be increased, by reducing the number of molecules per cm^3 volume. The molecules evaporate from the surface and travel few cm without colliding with the molecules of the residual gas in the space above. If now the condensing surface is placed within distance, a major fraction of the molecules will condense and will not return to the distilland. Thus each molecule distills itself and hence called "*Molecular Distillation*".

Characteristics of molecular distillation

1. Molecules having molecular weight within the range of 300 to 1100 dalton can be distilled by this method. [N.B. Low molecular weight (below 300 dalton) molecules will re-evaporate

again from the condenser surface. High molecular weight molecules (greater than 1100 dalton) will not have sufficient volatility.]

2. The molecules to be distilled should reach the surface and evaporate. The molecules at the bottom of the distilland have to overcome the pressure of the layer above, to come to the surface. Hence, the layer should be thin and should be in a state of turbulent motion to facilitate the molecules to reach the surface.
3. The distilland should be degassed before entering in the still, because at very low pressure the dissolved gas will occupy all the space and rate of distillation will be reduced.

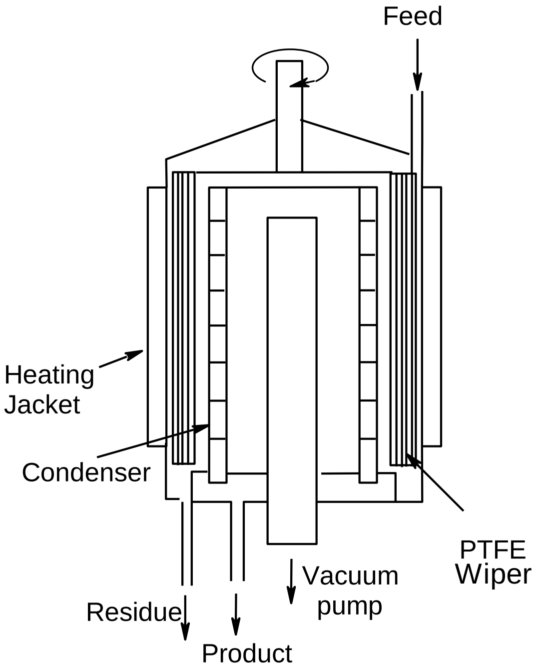


Fig. Falling film Molecular Still

Falling Film Molecular Still

The vessel has a diameter of the order of 1 m and the walls are heated suitably by a heating jacket. Vacuum pumps are connected by a large diameter pipe. The feed flows down the walls and is spread to a film by the polytetrafluoroethylene (PTFE) wipers which move about 3 m/s giving a film velocity of about 1.5 m/s. The residue is collected at the bottom of the vessel and it is re-circulated (through the feed line).

The evaporated molecules are then condensed on the condenser surface. The condensate is taken out as product.

Centrifugal molecular still

The distilland (feed) is introduced on to the center of a bucket-shaped vessel (1 to 1.5 m in diameter) that rotates at high speed. The film of liquid that is formed moves outwards over the surface of the vessel to the residue-collection pipe. The vessel is heated by radiant heaters. Condensers and a collection device are located close to the inner surface of the rotor.

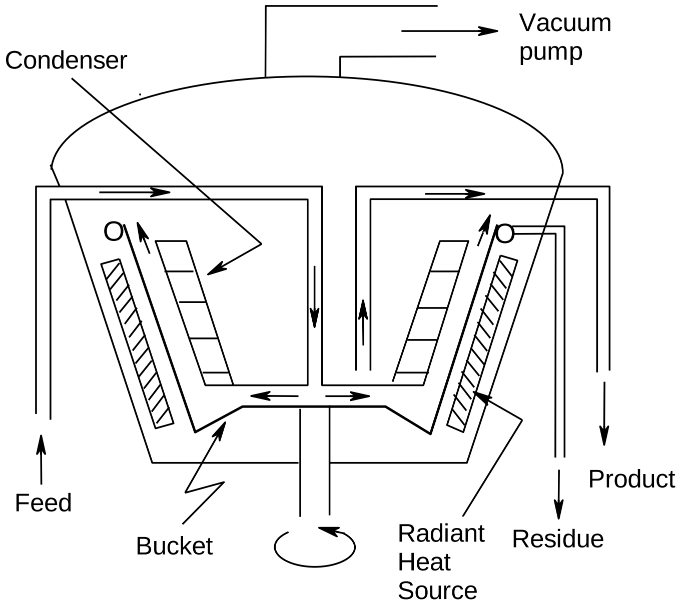


Fig. Centrifugal molecular stilll

Application of vacuum distillation in pharmacy

1. Vitamin concentrates

Vitamin A,D,E,K and tocopherols are obtained from vegetable and fish oils. The vitamin-A concentrate produced by molecular distillation is very pure and has good stability. As no chemical is used in this method which could split the ester linkage, the vitamins are retained in the natural ester form which is the most stable form of vitamin A. The stability of the concentrates is further enhanced by natural antioxidants distilling over from the original oil.

2. The fractionation of oil

The fractionation of oils into various components is carried out by molecular distillation.

Components	*Molecular Weight*	*Temperature Range*
(a) Fatty acids, unsaponifiable matter of low molecular weight.	150 – 300	50 – 140^0C
(b) Unsaponifiable matter like sterols, vitamins, dyes, waxy alcohols, monoglycerides	300 – 600	150 – 190^0C
(c) Triglycerides, sterol esters, vitamin esters, resins, waxes	600 – 900	Above 190^0C

3. *Purification and fractionation of lanolin*

It is used to get various fractions from Lanolin like, cetyl alcohol, cholesterol, ceryl alcohol, lanopalmitic acid,isocholesterol etc.

4. *Separation of Poly Ethylene Glycol (PEG)*

On laboratory scale it is used to separate PEG according to the degree of polymerization.

5. SPECIAL DISTILLATION METHODS FOR NON-IDEAL MIXTURES

Industrial scale distillation of Azeotropic Mixture

The liquor from fermentation process is a common source of ethanol and contains approximately 8–10% ethanol.

After simple distillation an azeotrope will form containing 95.6% (96E+4W) ethanol and boiling at 78.15^0C at atmospheric pressure.

In this type of system a reboiler is used instead of boiler. The feed liquor is introduced into the system and must occur at a point where the equilibrium will not be disturbed. Hence, feed will take place, at a place part of the way up the column, where the equilibrium composition on the plate is similar to the feed composition.

The plate below the *feed plate* form the stripping section where the rising vapor strips the more volatile component (ethanol) from the feed liquor while the upper section is known as the *rectifying section*.

The binary azeotrope produced at this stage is freed from water by making use of ternary azeotrope – ethanol, benzene, and water.

The ethanol/water azeotrope, with sufficient benzene (only required at start-up) is fed to column A and the pure ethanol is obtained as bottom product, since the ternary azeotrope takes off the water.

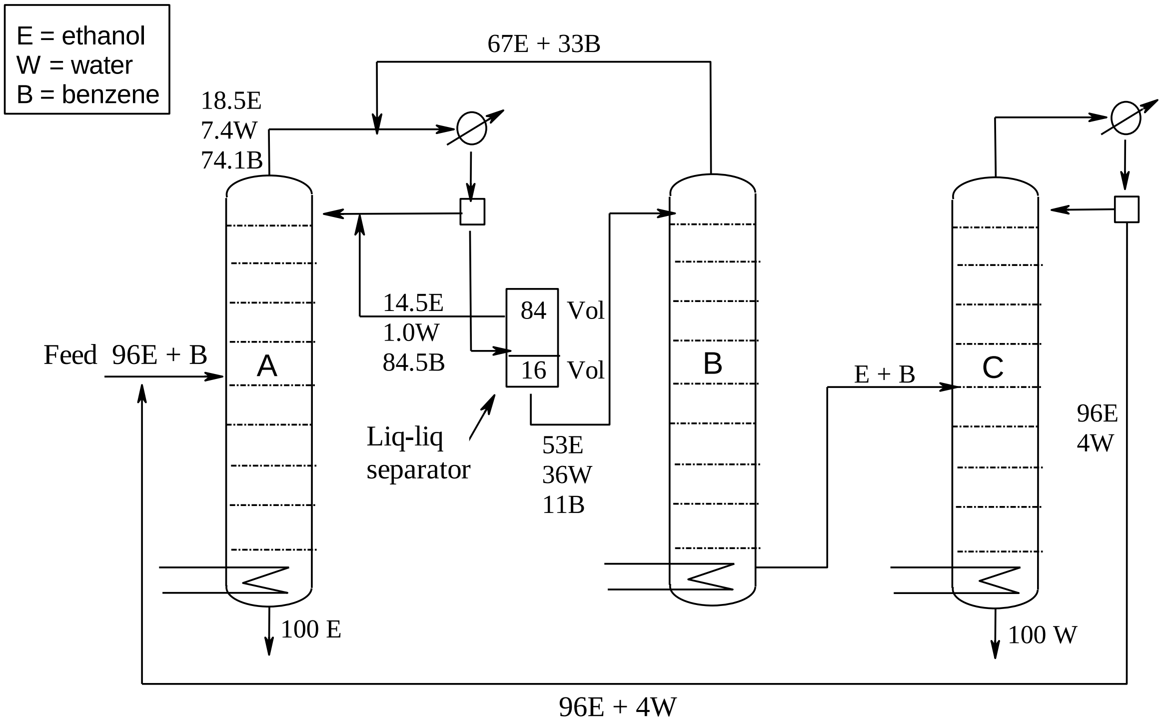


Fig. Plant for manufacture of Absolute ethanol (100% ethanol)

- The azeotrope (E+B+W) is taken from the top of the column A, condensed and separated (in liquid-liquid separator) into two layers, having the compositions given in the diagram.
- The upper layer predominates and, being rich in benzene (14.5E+1.0W+84.5B), is returned to column A. The lower layer (53E+36W+11B) is taken to column B, where the benzene is recovered as the ethanol/benzene binary azeotrope (67E+33B) and is mixed with the vapor from ethanol.

- The ethanol / water residue passes to column C, where the ethanol is recovered as the ethanol/water binary azeotrope (96E+4W), which can be incorporated with the original feed.
- The final product from column A is 100% ethanol and from column C is 100% water.

6. DISTILLATION OF IMMISCIBLE LIQUIDS

Steam distillation

Steam distillation is used for the distillation of two immiscible liquids one of which is water.

Application:

(i) Separation of volatile oil e.g. eucalyptus oil, rose oil, clove oil etc. and
(ii) Preparation of some aromatic water e.g. concentrated rose water..

Theory

Volatile oils are mixtures of high molecular weight compounds having low vapour pressure (i.e. high b.p.). To separate these from the natural sources like petals of flowers, barks etc. it is not possible to take them to their boiling points around 200^0C. If these oils are distilled with water (low molecular weight but high vapour pressure i.e. low b.p.) then volatile oil will be distilled out at a temperature below 100^0C.

$$\frac{\text{Weight of volatile oil in distillate}}{\text{Weight of water in distillate}} = \frac{M_V P_V}{M_W P_W}$$

Where, M_W and M_V are molecular weights of water and volatile oil respectively.

P_W and P_V are vapor pressure of water and volatile oil respectively.

- The aqueous phase of distillate that is collected is water saturated with volatile oil i.e. called *aromatic water*.

Absorption

In this process, a vapor in an inert gas is absorbed in a liquid stream. The vapor is soluble in the liquid. The liquid stream rich in solubilized vapor components is separated by distillation or some other suitable techniques. The liquid can be recovered and reused in the process itself. An example is removal of ammonia from ammonia – water mixture using water as solvent. Removal of carbon di oxide from a mixture of air and carbon dioxide using a solvent like primary or secondary or tertiary amines is another example. Typically this operation is carried out in counter current manner to enhance the efficiency of this process. Sometimes, the packed beds or staged beds like trays are used to have better mixing during the transfer process. It may be mentioned that absorption is a bulk phenomena, i.e., the gaseous solutes are transported to the bulk of the liquid stream. It may be noted that to effect separation, a matter is introduced in the system.

Adsorption

Separation of solutes by transporting it from gaseous or liquid streams on a solid surface is known as adsorption. As opposed to absorption it is a surface phenomenon. The solute is fixed on the solid surface only. Typically, the solid surface is specific for a particular component present in the feed stream. For example, in a liquid stream having organic as well as inorganic components, the organics are preferentially adsorbed on the surface of activated carbon. Typically, in a bed, the adsorbent particles are kept and the fluid is pumped through the system for intimate mixing. In the process, the solute is transferred to the solid phase. After sometime, the solid phase is completely saturated and the transfer of species becomes extremely slow due to lack of driving force. In such case, the bed has to be replaced by new adsorbents or the bed has to be regenerated by a suitable washing protocol. It may be noted, that a reasonably lower particle size of adsorbents provides sufficient surface area for separation. Removal of organics from an aqueous mixture using activated carbon is an example of adsorption. It may be noted that to effect separation, a matter is introduced in the system.

PHYSISORPTION

Molecules and atoms can attach to surface in two ways .

One is Physisorption (Physical Adsorption) in which there is a van der Waals interaction between the adsorbate & the substrate. Examples , A dispersion or dipolar-dipolar interaction . Van der Waals interactions are long range but are weak. Energy released when a particle is physisorbed is of the same order of magnitude as the enthalpy of condensation . Such small energies can be absorbed as vibrations of the lattice & dissipated as thermal motion, & a molecule bouncing across the surface will gradually lose all its energy & finally adsorb to it.

CHEMISORPTIONS

Chemisorption (Chemical Adsorption) in which the molecules or atoms stick to the surface of adsorbent by forming a chemical bond (usually covalent),& tend to find sites that maximize their coordination number with the substrate. The distance between the surface & the closest adsorbate atom is also typically shorter for chemisorption . A chemisorbed molecule may be torn apart at the demand of the unsatisfied vacancies of the surface atoms, & the existence of molecular fragments on the surface as a result of chemisorption is one reason why solid surface catalyse reactions

FACTORS AFFECTING ADSORPTION

TEMPERATURE

Adsorption is an exothermic process. Therefore in accordance with Le Chatelier's principle, the magnitude of adsorption should increases with decrease in temperature. It is in the case of physical adsorption . Chemical adsorption first increases with rise in the temperature and then starts decreasing

PRESSURE

With increase of pressure, adsorption increases up to certain extent till saturation level is achieved no more adsorption takes place no matter how high the pressure applied.

SURFACE AREA It's a surface phenomenon therefore adsorption capacity of adsorbent increases with increase in its surface area. Smaller the size of particles of solid adsorbents more is the extent of adsorption at its surface interface

ACTIVATION OF SOLID ADSORBENT Activation of adsorbent surface done so as to provide more vacant sites on surface. This can be done by breaking solid crystal in small pieces, breaking lump of solid into powders or sub- dividing the adsorbent

ADSORPTION ISOTHERMS

Adsorption process is usually studied through graphs known as Adsorption Isotherm. The amount of adsorbate on the adsorbent as a function if its pressure (gas) or concentration (liquid) at constant T

 According to Le Chatlier's principle, on application of excess pressure , the equilibrium will shift in the direction where number of molecules decreases.

 Since no. of molecules decreases in forward direction i.e.direction of Adsorption , with increase of pressure , forward direction equilibrium will be achieved

In the given Adsorption Isotherm , after saturation pressure Ps adsorption does not increases

 Vacancies on the surface of the adsorbent are limited, at Ps a stage is reached when all the sites are occupied and further increase in pressure does not increases adsorption extent.

Types of Adsorption Isotherms :

1- Freundlich Isotherm

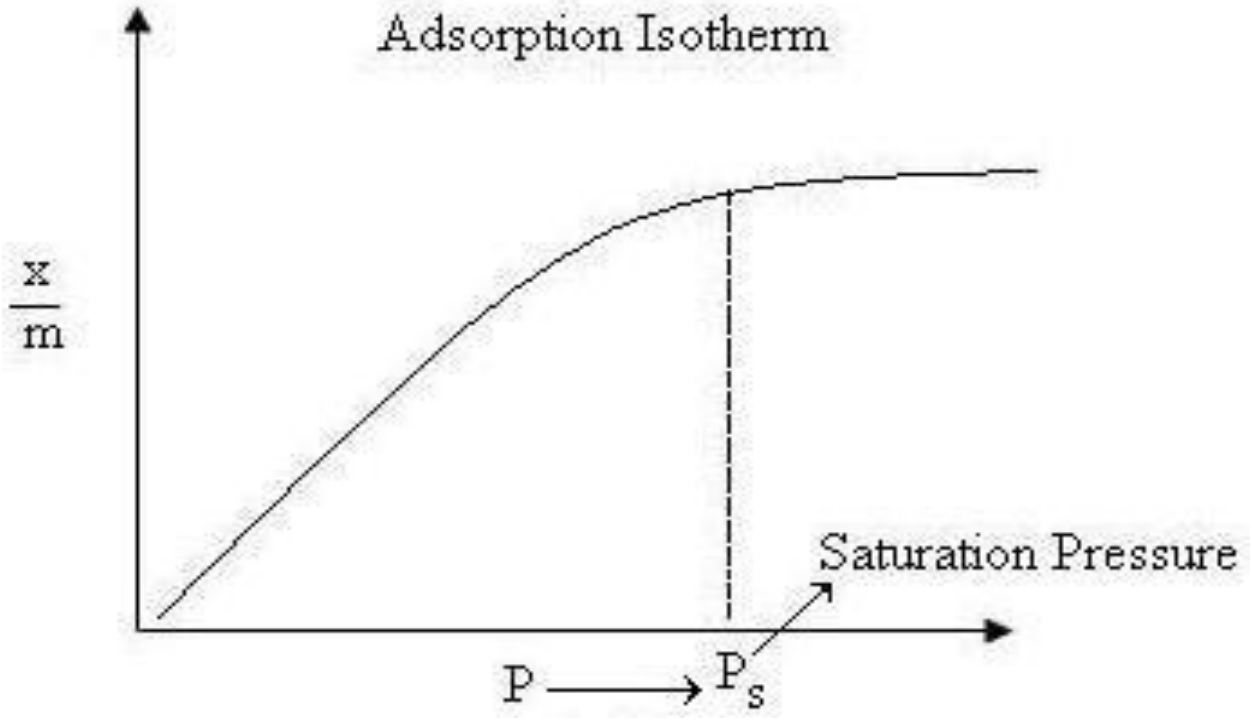
2- Langmuir Isotherm

3- Temkin Isotherm

4- Brunauer, Emmett And Teller (BET) Isotherm

- ***Equilibrium adsorption isotherm***
  - ***Adsorption isotherm relates the volume or mass adsorbed to the partial pressure or concentration of the adsorbate***

  - ***Quantity of adsorbate adsorbed (from gas phase) per unit adsorbent is measured against partial presence of the adsorbate (in gas phase) at equilibrium at given temperature (very sensitive to T)***
  - ***Incase of adsorbate in liquid/solution the measurement is against concentration of adsorbate in the solution***



Many equations are proposed to fit the experimental adsorption result

Which model/equation to be used to describe the adsorption process is determined through experimentation

BET (Brunauer,Emmett, and Teller) **Adsorption Isotherm**

$$\frac{V}{V_m} = \frac{cP}{(P_o - P)[1 + (c-1)(P/P_o)}$$

$$\frac{P}{V_{total}\left(P-P_0\right)}=\frac{1}{V_{mono}C}+\frac{c-1}{V_{mono}C}\left(\frac{P}{P_0}\right)$$

$$Y \quad = \quad C \quad + \quad m \quad X$$

V is the amount of adsorbed gas at a given pressure and temperature

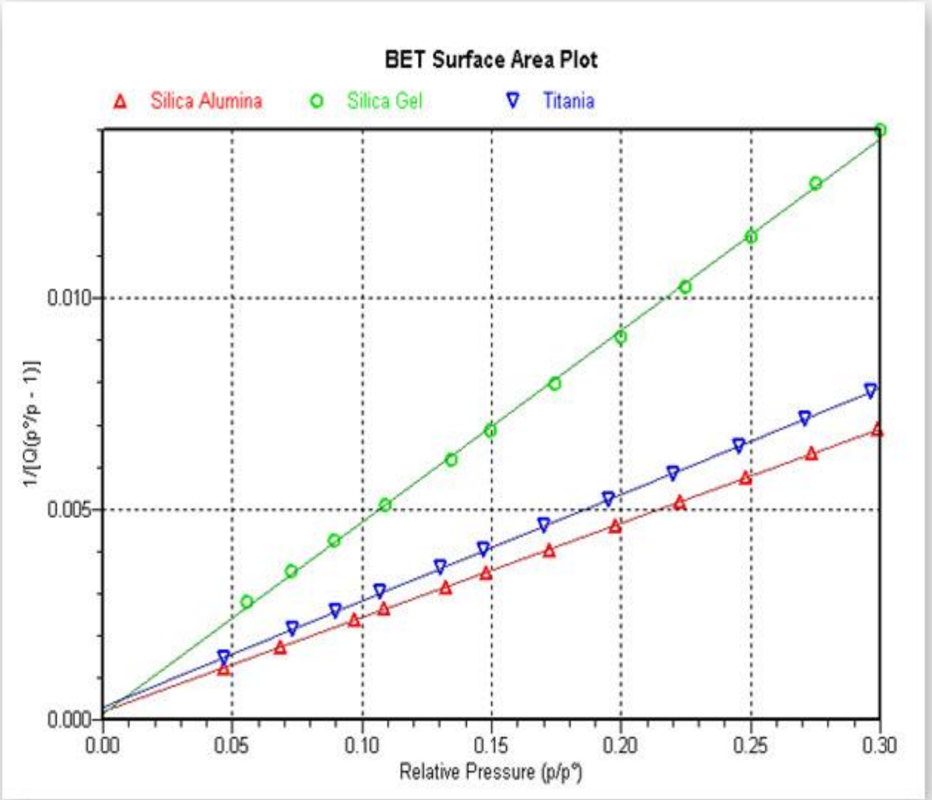
V_m is the amount adsorbed if one molecule thick layer fills the surface

P_o is vapor pressure of the adsorbate at the system's temp.

P is partial pressure of the adsorbate

C is a parameter of adsorption process

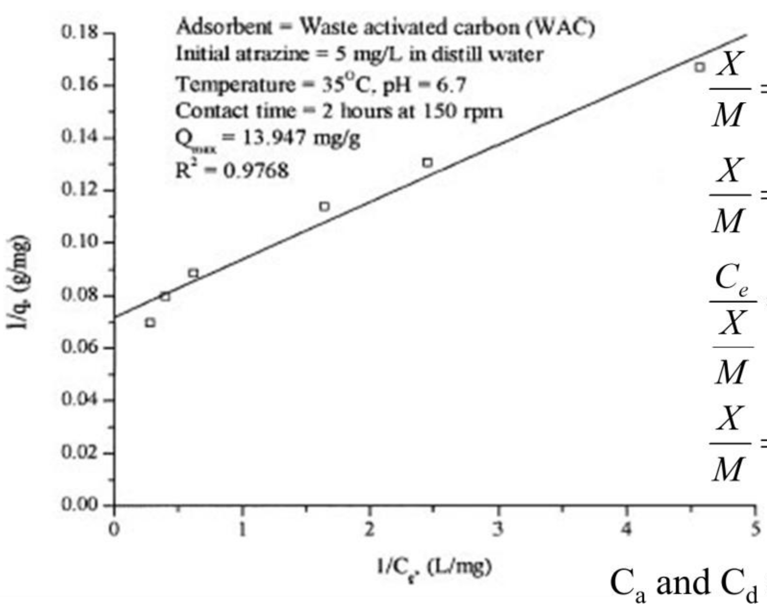
When P/P0 is <0.05 and >0.35 then the plot is not linear and this approach for estimating Vmono and C values can not be followed



Langmuir Adsorption Isotherm

Langmuir adsorption isotherm assumes unimolecular layer

Mass of adsorbate per unit mass of adsorbent is proportional to ‘f’



$$\frac{X}{M} = C_a^{'} f \quad where \quad f = \frac{C_a C_e}{C_a C_e + C_d}$$

$$\frac{X}{M} = C_a^{'} \frac{C_a C_e}{C_a C_e + C_d} = \frac{k_1 C_e}{k_2 C_e + 1}$$

$$\frac{C_e}{\frac{X}{M}} = \frac{k_2}{k_1} C_e + \frac{1}{k_1}$$

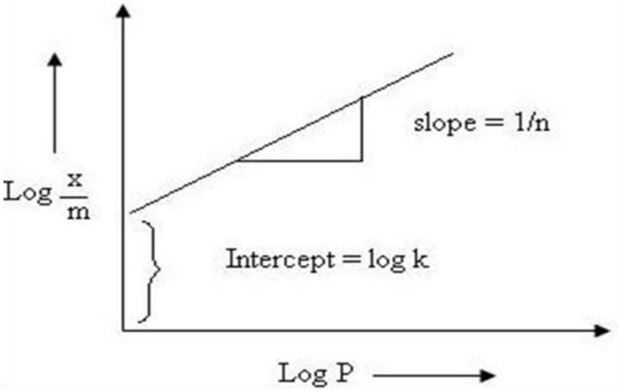
$$\frac{X}{M} = \frac{kbC_e}{1 + kC_e} \qquad \frac{M}{X} = \frac{1}{kb}\frac{1}{C_e} + \frac{1}{b}$$

C_a and C_d are constants

‘f’ occupied fraction of the total solid surface

C_e is concentration or partial pressure of the adsorbate

Fruendlich adsorption isotherm



$$\frac{X}{M} = kC_e^{1/n}$$

$$\log\left(\frac{X}{M}\right) = \frac{1}{n}\log C_e + \log k$$

k is freundlich capacity factor

n is Freundlich intensity parameter

Adsorption isotherms can also be constructed for heterogeneous mixture of compounds

Parameters, like, TOC, COD, dissolved organic halogens, fluorescence, UV absorbance, are used to measure adsorbate concentration

Temkin Isotherm

Temkin Isotherm takes into account all the indirect adsorbate adsorbate interactions on adsorption isotherms

$$\theta = c_1 \ln (c_2 p)$$

.... **TEMKIN ISOTHERM**

where c1 and c2 are constants, corresponds to supposing that the adsorption enthalpy changes linearly with pressure Drying

In drying, water vapors from a solid material are removed by using a vapor stream to an acceptable small limit. Generally it is carried out by applying thermal energy as compared to introduction of a matter in case of absorption or adsorption. The process efficiency depends on the temperature, relative humidity and other thermo physical properties of the drying vapor as well as the temperature and other thermo-physical properties of the solid.

RAATE GOVERNED PROCESS:

Most of the membrane based processes are the rate governed. The main crux of the separation lies in the difference of transport of various species through the membrane. The driving force of such transport is generally gradient of chemical potential. If we recall the definition of gradient of chemical potential, it is composed of four parts. These are concentration gradient, pressure gradient, temperature gradient and electrochemical potential gradient. Presence of these causes (one or more of such gradients) results into effects (the difference in separation and hence effects). Some of these systems are described below.

Osmosis

Osmosis is observed when a solution is separated from the solvent by a semipermeable (only solvent is permeable species) membrane. Apparently, difference in osmotic pressure across the membrane causes the transport of the solvent. Actually, osmotic pressure is a colligative property and it is a function of concentration. Therefore, the concentration (or more generally activity) gradient of the solvent is the main driving force in osmosis. In this case, the solvent flows from the solvent side to the solution side until, the solvent activity becomes almost equal in both the sides. This results in generation of hydrostatic pressure difference between the two sides, known as osmotic pressure. If the initial concentration of solute is more in the solution side, more solvent will flow in from the solvent side to equalize the solvent activity on both the sides, resulting in more osmotic pressure difference.

Reverse Osmosis

It must be understood that osmosis is a natural phenomenon. In case of reverse osmosis, pressure is applied on the solution side externally (using a pump or a compressor), so that the osmotic pressure is overcome and the solvents are forced out of the solution side. The efficiency of this process depends on the porosity, morphology and thickness of the membrane. These factors influence the transport of the solvent through the membrane immensely and thereby, dictating the throughput of the process and the quality of the product. Since, reverse osmosis membranes have very small pore size, this is used for separation of lower molecular weight species, like, salts. A salt having very osmotic pressure, the operating pressure in the feed side of a reverse osmosis process is therefore extremely high. This will lead to deposition of salts near the membrane surface, leading to build up of a concentration boundary layer. Therefore, the pressure gradient in this system leads to concentration difference.

Dialysis

In this case, two liquid streams are separated by a permeable barrier or membrane. In the feed side, a specific set of solutes are permeated through the membrane to the other side. The upstream feed is known as the feed side and the downstream is known as the dialysate. Typically, dialysate stream is pure distilled water. Thus, the concentration gradient between the two streams is the maximum. The transport is effected by the concentration gradient between two streams. The duration of separation entirely depends on the rate

Ion exchange

Is defined as the process where an insoluble substance remove ions of positive or negative charge from an electrolytic solution and releases other ions of the same charge into the solution in a chemically equivalent amount. That means here exchange of ions will be taking place. So one type of ions will be getting removed from the solution but equivalent amount or equivalent amount of ions will be coming from the resin that is why it is known as ion exchange process

THEORY OF EXTRACTION OF DRUGS

Mass transfer

Consider a crystal of soluble material (e.g sugarcube) is immersedin asolvent in which it is dissolving. The crystal will be surrounded by a stationary boundary layer of the solvent, with the bulk of the fluid able to move.

The transport of molecules will take place in two stages:

1. The molecules will move through the boundary layer by **molecular diffusion**, with no mechanical mixing or movement.
2. Once material has passed through the boundary layer, mass transfer takes place by bulk movement of the solution, known as **eddy diffusion**.

Since there is no limit to the vigor of the movement of the bulk of the fluid, so the **rate controlling step** is the molecular diffusion.

Mass transfer by molecular diffusion can be represented by an equation, similar to conduction of heat transfer, in which

$$\frac{m}{t} = \frac{DA(C1 - C2)}{h}$$

where m = mass transferred in time t

D = diffusion coefficient of the solute

A = area of the solute exposed to the solvent

C1 = concentration of the solute at solid / liquid interface

C2 = conentration of solute in the bulk phase

h = thickness of the stagnant layer

Theory of extraction of drugs

Examination of the extraction processes will show that all have certain stages in common:

(i) Suitable size reduction of the drug
(ii) Penetration of the drug by the solvent
(iii) Solution of the soluble material within the cells.
(iv) Escape of the soluble material through the cell walls and through the solvent boundary layer surrounding the particles of the drug.
(v) Separation of the solution and the exhausted drug.

(I) Suitable size reduction of the drug:

From the mass transfer equation it is evident that m/t is proportional to A. If the area (A) is increased then rate of dissolution also increases. So if the size of the drug (plant or animal parts containing the active constituents) is reduced surface area will increase, the consequence of which is the increase of the dissolution rate (m/t) of the active constituent. The **ideal size reduction** would be at cellular level but it poses the following disadvantages:

(i) It is difficult to reduce the size of a drug to its cellular dimension. It requires costly instruments which may not be cost effective.
(ii) Obviously it will take more time to reduce the size to such a level. Thus prolonged comminution (size reduction) will produce heat that may damage the heat labile constituents of the drug.
(iii) After extraction of such small particles they will make a suspension which will be difficult to filter.
(iv) While reducing the size to cellular level it is most probable that the cell walls will be broken and breakage of cell walls will release unwanted cellular materials like gums, starch, proteins etc. which may produce the filtrate cloudy by the release of **colloidal material**.

So the degree of size reduction to be used will depend, therefore, on the botanical structure of the drug.

(II) Penetration of the solvent into the drug:

Before drying the fresh drugs are surrounde by a thin film of water. After drying that water film evaporates and becomes porous due to shrinkage. The pores are then occupied by air. To penetrate the cell wall the solvent must have to displace the air first.

When the dry drug is moistened, the liquid film is again renerated and then the cells imbibe the solvent and swell.

Sometimes to facilitate the removal of air from the pores the solvent and the drug is first taken in a vessel, vacuum is applied - thus air is removed from the pores. Then, when the vacuum is released, pressure of the atmosphere forces the solvent into the drug and penetration is facilated considerably.

(III) Solution of constituents:

Once the solvent has penetrated into the cells solution of the constituents takes place and is governed by the solubility of the constituents in the solvent and again solubility depends on the temperature. So if the temperature is increased the solubility will also enhance.

(IV) Escape of the solution from the cells:

The solute molecules are transferred through the boundary layer or stagnant layer. Factors controlling mass transfer will show that the rate of extraction can be affected in the following ways:

(i) $m / t \propto 1 / h$ i.e. if the thickness of the boundary layer can be reduced rate will ne increased. h can be reduced by agitating the mixture occassionally which disperses local concentrations of the solution, thereby, increasing the concentration gradient.

(ii) By suspending the drug in a cloth bag or placing it on a perforated plate near to the surface of the liquid the escape of the solution can be hastened. As the constituents dissolve, the density of the solution increases, so that convection currents are established, leading to circulation of the solution followed by the reduction of local concentration surrounding the cloth.

(V) Separation of solution and exhausted drug:

After dissolution the solid materails has to be strained off. Since, the drug absorbs solvent and there is a residue of soluble constituents in that solvent, so the drug is subjected to pressure and sometimes under hydraulic pressure.

PROPERTIES OF SOLVENTS USED FOR EXTRACTING DRUGS

The ideal solvent would be:

1. It must be cheap.
2. Non-toxic
3. It should be stable chemically and physically.
    a) neutral to reaction
    b) not too volatile
    c) non-inflammable
4. Selective, i.e. it should remove the desired constituents with minimum amount of the inert materials.

Water as a solvent for extraction:

Advantages:

(i) It is cheap.
(ii) It has a wide solvent action (e.g. protein, coloring maters, gums, antharaquinone derivatives, most alkaloidal salts, glycosides, sugars and tanins).
(iii) It is non-toxic and can be taken internally.

It is non-inflammable. In industry handling of large volume of volatile solvents may cause accident. Hence the inflammability of a solvent is very important from the point of view of industry.

Disadvantages:

(i) It is not selective. It dissolves a wide range of substances that may interfere with the extract's clarity e.g. gum, protein (coagulated).
(ii) Water is good medium for mold and bacterial growth. Generally most preparations are preserved with a small amount of glycerine, chloroform or by sterilization.
(iii) Water promotes hydrolysis of many substances and allows enzymatic actions to take place [e.g glycosides such as digitalis].
(iv) Concentration of aqueous solution requires more heat than for most other solvents due to its higher latent heat of evaporation (537 cal / gm).

Ethanol as solvent:

Advantages:

(i) Reasonably selective, e.g. in a drug containing gum, albuminous matter and a glycoside or an alkaloidal salt, ethanol in a suitable dilution with water would dissolve only the glycoside or the alkaloidal salt, whereas water would usually dissolve all of the constituents.
(ii) Molds cannot grow in solvent mixture containing more than 20 % ethanol.
(iii) Non-toxic in the quantities prescribed in the medicinal preparations.
(iv) It is neutral, hence compatible with other products .
(v) Latent heat of vaporization is less than water , so less heat will be consumed to make an extract concentrated.
(vi) Can be mixed in any combination with water.

Disadvantage:

Cost due increases due to the duty imposed by the Government on it

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UNIT – 3 BASIC SEPARATION PROCESSES-2 SCHA1311

UNIT 3

BASIC SEPARATION PROCESSES – 2

Drying, -Tray dryer, Vacuum dryers, rotary dryers, drum and spray dryers. Equilibrium moisture content of materials. Bound and unbound moisture. Free and equilibrium moisture, Rate of Drying curves, Freeze drying and Sterilization of Biological materials, Crystallization theory, Equipments for crystallization, Tank, DTB Crystallizer, circulating –magma vacuum crystallizer, Swenson walker crystallizer.

Drying is defined as the removal of liquid from a solid by thermal method.

When large amount of liquid is evaporated from a solution /suspension / slurry the process is called 'evaporation'. The final product is a concentrated liquid / slurry.

When very small amount of liquid is evaporated from solids the process is called 'drying'. The final product is a 'dried solid'.

PURPOSE OF DRYING

1. Drying is most commonly used in pharmaceutical industries in the preparation of granules, which can be packed in bulk or compressed into tablets or filled in capsules.
2. Drying is required for processing of materials like, drying of aluminium hydroxide, spray drying of lactose and preparation of powdered extracts.
3. Drying is used to reduce the bulk weight that lowers the transportation and storage costs of that material.
4. Drugs obtained from plant and animal sources, when dried, becomes more friable. Thus drying helps in size reduction of natural drugs.
5. Animal and vegetable drugs are preserved against mold growth in dried condition.
6. Dried products often are less more stable than moist ones as in the case of effervescent salts, aspirin, hygroscopic powders, ascorbic acids and penicillin.

SOME DEFINITIONS

Moisture content (MC)

$$\%\ \text{Moisture Content} = \frac{\text{weight of water in sample}}{\text{weight of dry sample}} \text{ x } 100$$

Loss on drying (LOD)

$$\%\ \text{LOD} = \frac{\text{weight of water in sample}}{\text{total weight of wet sample}} \text{ x } 100$$

THEORY OF DRYING

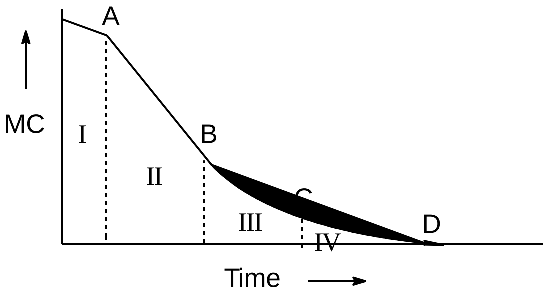
A typical drying cycle shows the following phases:

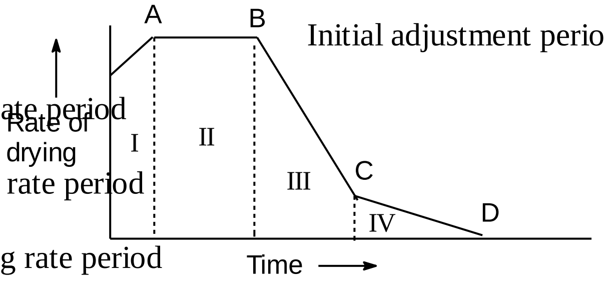
Phase-I: Initial adjustment period

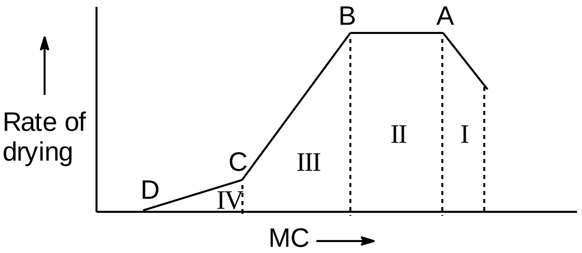
Phase-II: Constant rate period

Phase-III: First falling rate period

Phase-IV: Second falling rate period







B: Critical Moisture Content (CMC)
D: Equilibrium Moisture Content (EMC)

The rate of drying of a sample can be determined by suspending the wet material on a scale or balance in a drying cabinet and measuring the weight of the sample as it dries as a function of time.

Phase-I: Initial adjustment period

A wet material when kept for drying it begins to absorb the heat and the temperature of the material increases. At the same time, the moisture begins to evaporate and thus cools the drying solid. After a period of initial adjustment, the rates of heating and cooling become equal and the temperature of the drying material reaches the *wet-bulb* temperature of the *drying air*.

Phase-II: Constant rate period

During this period there is continuous liquid film over the surface of the solid. Evaporation from the film (at wet bulb temperature) proceeds at a constant rate and the film is continuously replaced by the underlying moisture. As long as the delivery of water from the interior of the material is sufficient to keep the surface completely wet, the drying rate remains constant.

Drying rate at this phase is given by:

$$\frac{dW}{d\theta} = \frac{Ah_c\,(t_d - t_w)}{\rho L \lambda}$$

where, $\frac{dW}{d\theta}$ = Rate of drying in $\left(\frac{\text{kg of water}}{\text{kg of dry solid}}\right)$

A = Area exposed to drying

t_d, t_w = Dry bulb and wet bulb temperature of the drying air respectively

ρ = Bulk density of solid

L = Thickness of solid bed

λ = Latent heat of vaporization of water

h_c = Convection heat transfer coefficient

Phase-III: First falling rate period

Due to dry spots on the surface, the area of constant mass transfer decreases and the heat transferred to the dry spots will be utilized to raise the temperature of the material to the dry bulb temperature. Thus as the number of dry spots increase, heat transfer and mass transfer rates fall which is called the first falling rate period. The point from which Phase-II starts is called **critical point** (B). Moisture content at which this point appears is called **critical moisture content (CMC)**.

Phase-IV: Second falling rate period

In this period the capillaries are empty, no film is present on the surface. The moisture movement takes place only by diffusion. In this phase the rate of drying falls and it is lesser than first falling rate. The starting of second falling rate period is called **second critical point** (C).

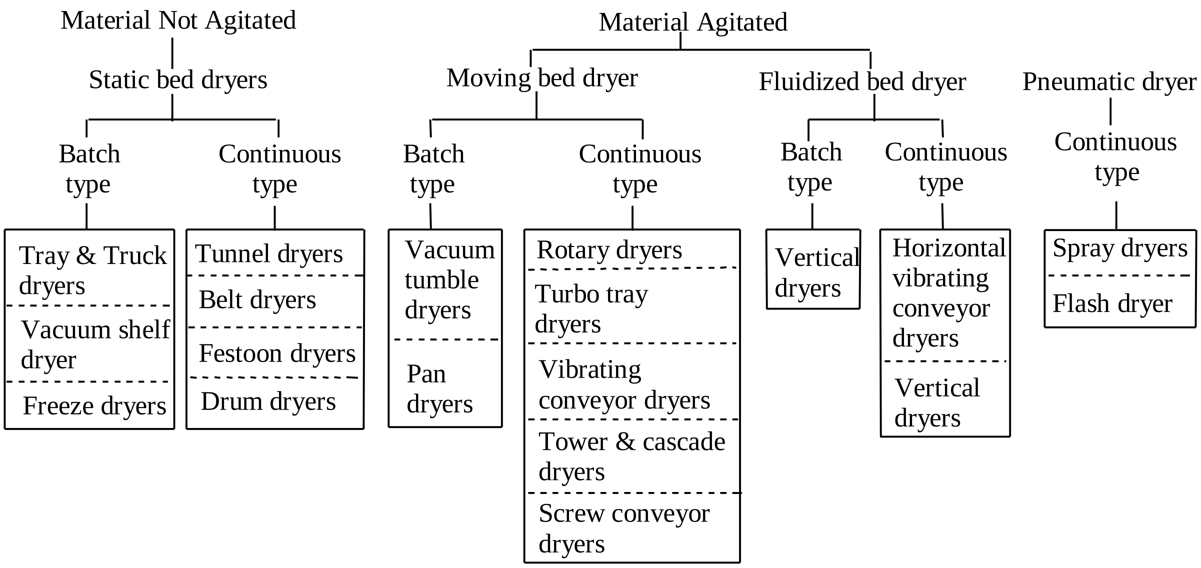
At the end the drying rate becomes zero. The moisture content at this point is called **Equilibrium Moisture Content** (EMC). EMC may be defined as the *moisture content of the solid when drying limit has attained by use of air at a given temperature (dry bulb) and humidity of air.* EMC depends on the *nature of the material, temperature* and *humidity*.

Utility of drying curve

1. From the drying curve the *time for drying* a batch of material in a certain dryer can be estimated.
2. The size of a particular dryer can be determined for drying a substance from one moisture level to the desired moisture content.

CLASSIFICATION OF DRYERS

Classification based on solid handling



Classification of dryers, based on methods of solids handling

Classification based on heat transfer mechanism

A. Convection dryers

(a) Tray or shelf dryers (b) Tunnel dryers(c) Rotary dryers (d) Fluidized bed dryers

B. Conduction dryers

(a) Vacuum oven (b) Freeze dryers

C. Radiant heat dryers

(a) Infra-red dryers

TRAY DRYER / TRUCK DRYER / SHELF DRYER / CABINET DRYER / COMPARTMENT DRYER

Construction

It consists of a small cabinet or a large compartment in which trays containing wet materials are placed. The compartment wall is insulated to reduce heat loss.

- In <u>tray dryers</u> the trays are directly placed inside the cabinet.
- The <u>truck dryer</u> the trays are loaded on to the trucks (shelves on wheel) and then the trucks are introduced inside the heating cabinet.

The bottom of the trays are either perforated or having wre-mesh bottom.

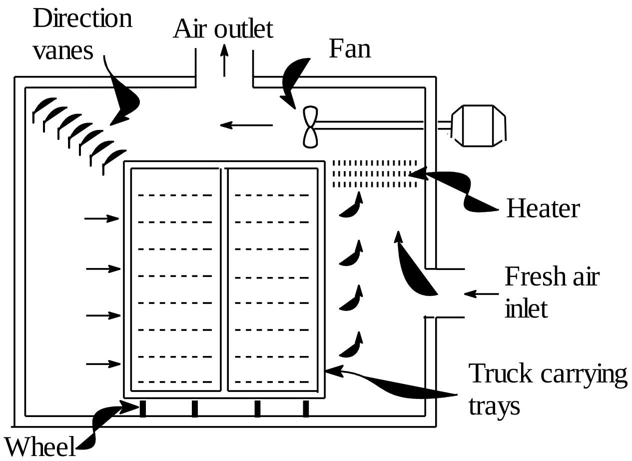


Fig. Truck dryer

- The material is heated by hot air circulated by means of fans that removes the humid air from the cabinet.

The trays containing the load remain in the dryer until drying is complete, after which they are withdrawn, emptied and recharged for drying the next batch.

<u>Energy sources</u>: Dry air can be heated either by electricity or steam.

Applications

- Drying of crude drugs, chemicals, powders, tablet granules etc.
- It is a batch process and materials can be handled separately.

ROTARY DRYERS

Construction

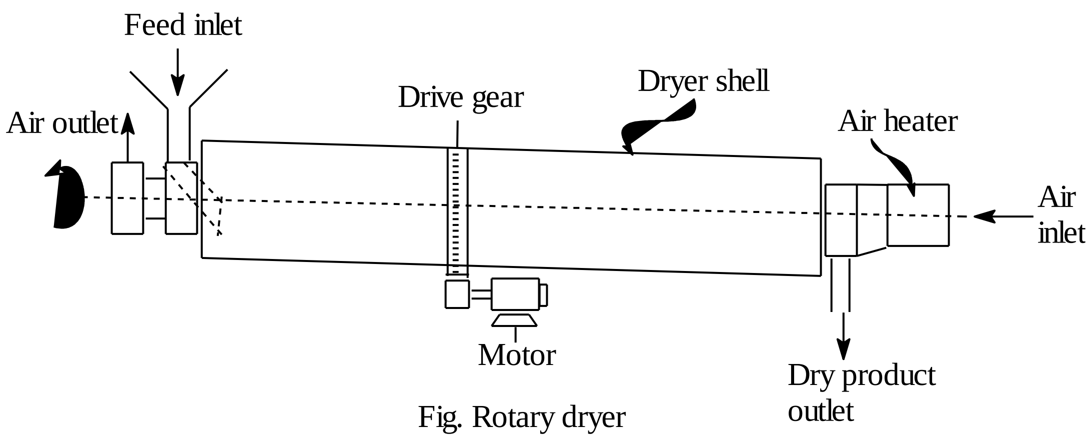


Fig. Rotary dryer

It is a cylindrical shell (10 m length) mounted with a slight slope so that the material will move through the shell as it is slowly rotated at about 10 rpm. To improve contact the shell contains baffles or flights, which lift the solids and spill the particles through the air stream.

The hot air flows counter current to the flow off material.

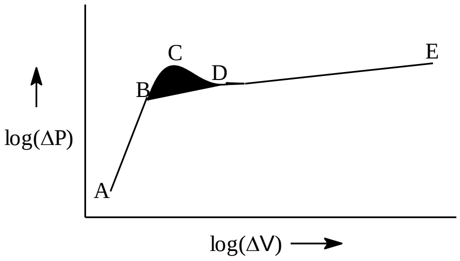
Application

It is used for continuous drying on a large scale of any powdered or granular solid.

FLUIDIZED BED DRYER

Principle

Let us consider a situation where a bed of granules is placed over a perforated bottom container and hot air is flown from bottom through the bed. The pressure drop (ΔP) across the bed and the air velocity (V) are measured. If the air velocity is gradually increased and ΔP is plotted against V then the following curve is observed.



- *Point A*: When the air velocity is very low flow takes place between the particles without causing any disturbance.
- *Point B*: When the velocity is increased to a certain value the frictional drag on the particles become equal the force of gravity of the particle.
- *Point C*: Rearrangement of the particles occurs to offer least resistance.
- *Point D*: Eventually the particles are suspended in the air and can move, ΔP decreases slightly because of greater porosity.
- Further increase in the air velocity causes the particles to separate and move freely, and the bed is fully *fluidized*. Any additional increase in velocity separate the particles further, i.e. the bed expands, without appreciable change in ΔP until E.

- In the D-E region the air flows through the bed in the form of bubbles – the term *boiling bed* is generally used for this stage.
- Above point E the solid particles entrain into the gaseous phase and the particles float in the gas.

Construction

Two types of fluidized bed dryers are there

(i) vertical fluidized bed dryer – for batch process
(ii) horizontal fluidized dryer – for continuous process

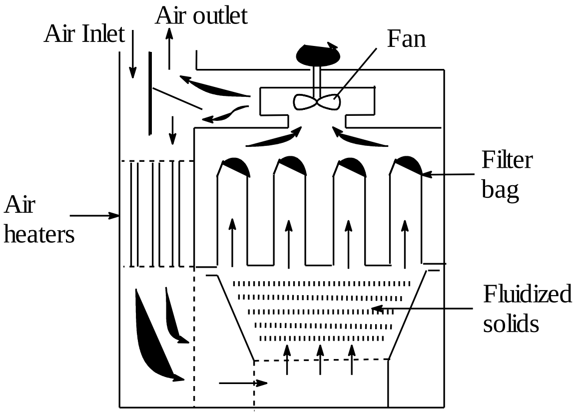


Fig. Fluidized bed dryer

The dryer consists of:

(a) Air handler: This is a source of dry and hot air. It is also attached by means of heating and dehumidifying air, if necessary.
(b) Plenum: It consists of a screen or plate to distribute the incoming air as it enters the dryer.
(c) Product container: This container holds the product that is to be dried.
(d) Expansion chamber: This chamber is situated above the product container and holds the suspended material.
(e) Filter: The upper part of the expansion chamber has bag filters. It prevents fines from escaping into the atmosphere or collecting on the blades that pulls the air through the dryer.

Applications

1. Wet granulation:

(a) Fluidized bed dryers are used to dry the previously prepared wet granules.
(b) Powders are agglomerated in the drying chamber by spraying liquid binder over it, while the hot air dries the agglomerates to form dry granules.

2. Coating of tablets

The fluidized bed dryer can be used for coating granules also. This technique is called Wurster technique. Three basic designs are employed

(a) top spray(b) bottom spray(c) tangential spray

In fluidized condition the powder is coated by coating solution sprayed from the nozzles. As the particles are coated they become heavier. When the mass developed becomes higher than the drag force given by the fixed air velocity the particles no longer floats. They fall back, which is then collected as product.

Advantages

1. Efficient heat and mass transfer facilitate high drying rates. Heating time of thermolabile materials is minimized.
2. Individual particles of the bed get dried in the fluidized state. So, most of the drying will be at constant rate and the falling rate period is very short.
3. Temperature can be controlled uniformly.
4. A free-flowing product is obtained.
5. Since the bed is not static, free movement of individual particles eliminates the risk of soluble materials migrating.
6. Short time yields a high output from a small floor space.

Disadvantages

1. Turbulence of fluidized state may produce fine particles due to attrition.
2. Fine particles lead to segregation, so they must be collected by bag filters.
3. Static charges may be produced due to vigorous movement of particles in hot dry air.

VACUUM DRYER

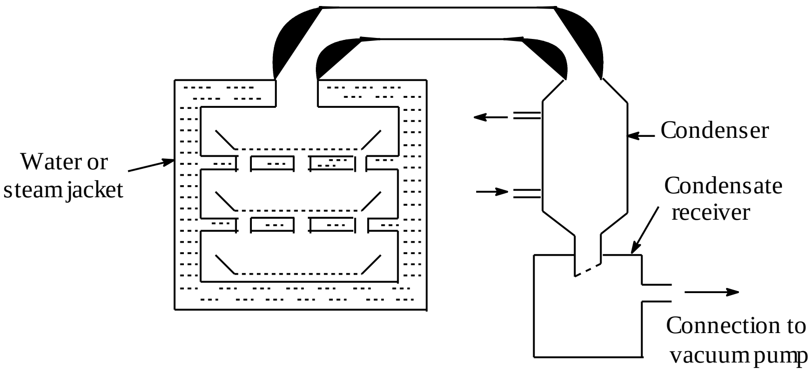


Fig. Vacuum dryer

Conduction is used as the principle method of heat transfer in dryers that are operated under vacuum. Convection cannot take place when air is nearly absent.

Construction

It is a jacketed vessel through which steam or hot water is passed. The vessel can be closed airtight. The oven is connected through a condenser and receiver to a vacuum pump. The supports of the shelves form part of the jacket, giving a larger area for heat conduction. Materials to be dried are kept in a tray and placed on the shelves. Hot water or steam is passed through the jacket, a vacuum pump is connected to the chamber.

Advantages:

1. Drying takes place at low temperature, so thermolabile materials can be dried.
2. It reduces the risk of oxidation during drying.
3. It produces porous and friable granules. [N.B. Because under vacuum the vapor forms bubbles and in this condition the material is dried.]
4. The solvent can be recovered from the condenser.

Disadvantages

1. Heat coefficients are low. Most of the heating takes place by conduction, some is from radiation from the wall of the jacket around. So the drying rate is slow.
2. Labor and running costs are high.

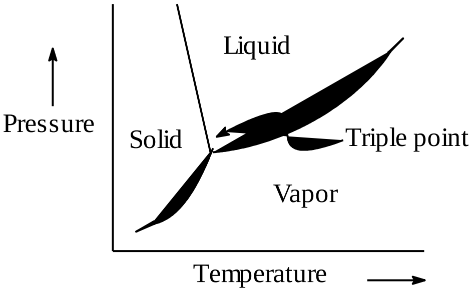
Applications:

1. To dry a thermolabile material like Penicillin.
2. To produce porous form such as dry extract.
3. To recover the solvent, for example to recover ethanol from ethanol extractives.

FREEZE DRYER

Principle

The temperature and pressure of the material is reduced below the triple point of solvent to be dried. Under these conditions, any heat transferred is used as latent heat and the ice sublimes directly to vapor state (without formation of liquid state).



Triple point of pure water is 4579 μm of Hg and 0.0099^0C. Pharmaceutical products remain in solution. In this case the pressure and temperature below which water evaporates directly from ice to vapor state is called *eutectic point*. In freeze dryer the pressure and temperature is maintained well below the *eutectic point*. Generally it is carried out at -10^0C to -40^0C, and at pressure of 2000 to 100 μm Hg.

Construction

Freeze dryer consists of

1. *a chamber for vacuum drying*:

   Two types of chambers are there, one for batch type and another for continuous type operation.

2. *a vacuum source*:

   Vacuum is achieved either by vacuum pump or by steam ejector or a combination of two.

3. *a heat source*:

   Heat is provided by conduction or radiation.

4. *a vapor removal system*:

For removal of water vapor condensers, desiccants, pumps or scrapper blades are employed.

Stages of freeze drying process

(a) Preparation and pretreatment:

Protein solutions take 8 to 10 times longer period than pure water. Therefore, in such cases, it is desirable to concentrate the solution under normal vacuum tray dryer.

(b) Pre-freezing

The aqueous solutions to be dried are packed in vials, ampoules or bottles. They are then cooled to solidify the water. Cooling can be done by using cold-shelves (–50^{0}C), alcohol baths (–50^{0}C) or liquid nitrogen bath (–195^{0}C).

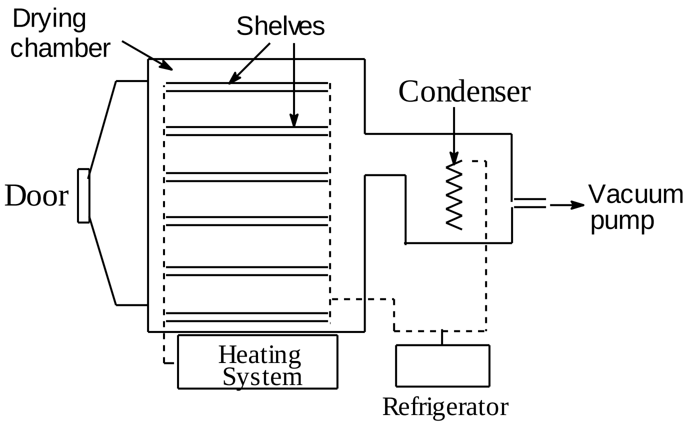


Fig. Industrial freeze dryer

- Thinner the layer of frozen material higher is the drying rate. The usual thickness is kept at 0.5 to 0.75 inches.
- Low freezing rates produces larger crystals of ice. Sublimation of water from this material leaves large pores. So freezing rate is generally maintained at 3 to 25^{0}C/min resulted in a product having pore size of 1 to 45 μm.

(c) Primary drying (Sublimation of ice under vacuum)

A vacuum of 0.5 bar is applied on the frozen materials. The temperature is increased to 30^{0}C within 2 hours. Then the temperature is kept constant. During this stage around 98 to 99% water is removed from the materials.

(d) Secondary drying (Removal of residual moisture under high vacuum)

Temperature is maintained at 30^{0}C continuously and vacuum is lowered to a pressure of 0.07 bar. The rate of drying is very low it takes 10 to 20 hours to dry 1% moisture.

(e) Packing

Inert gas is introduced inside the dryer to break the vacuum. Then the vials and ampoules are sealed within the dryer to reduce the contact of atmospheric gases.

Advantages

1. Drying takes place at a very low temperature, so that the enzyme action is inhibited, and decomposition (e.g. hydrolysis) is minimized.

2. The solution is frozen, so that the final dry product is a network of solid occupying the same volume as the original solution. Thus there is no case-hardening and the product is light and porous.
3. The dried products are readily re-dissolved or re-suspended by the addition of water prior to use (this procedure is termed as *reconstitution*).
4. The solutions do not concentrate during drying (like in other drying methods). Hence salts do not concentrate and denature the proteins present in the same solution.
5. Under high vacuum there is no contact with air, and oxidation is minimized.

Disadvantage

1. It produces a very hygroscopic product, hence should be sealed in the final package within the dryer.
2. The process is very slow.
3. The instruments are very costly.

Applications:

1. Maintenance and preservation of microbial culture.
2. Solution of penicillin can be stored at 0 – 2^0C and used within two-three days, but if freeze dried then it is stable for several months.
3. To produce fibrin foam [N.B. Fibrinogen is dissolved in sodium chloride injection and whipped into a foam that is then clotted by addition of human thrombin. The foam is then freeze dried].
4. To prepare gelatin sponge [N.B. A solution of gelatin containing traces of formaldehyde is foamed, freeze dried, sterilized and used as surgical dressing.]
5. Used to dry sera, blood products, certain enzymes, plant extracts, diagnostics, mammalian tissues useful in skin and bone graft surgery.

SPRAY DRYING

In spray dryer the liquid is sprayed in small droplets by atomizer in a drying chamber so that each droplet dries to a solid particle. The feed may either be solution, suspension or paste.

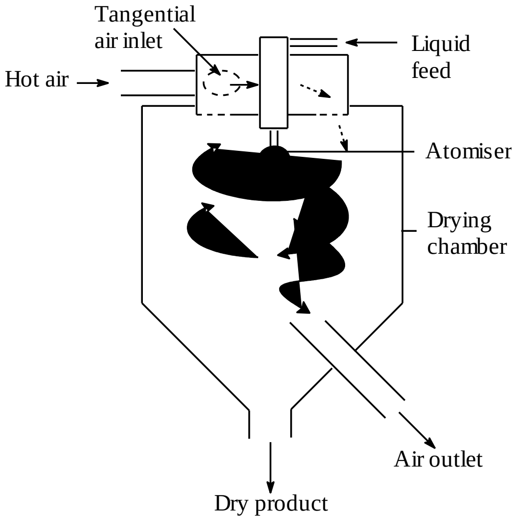


Fig. Spray dryer

Construction and working

The spray drying process involves four basic stages:

(A) <u>Atomization of feed into spray</u>

When a feed is atomized into small droplet its specific surface area increases many folds. So drying takes place at a very fast rate. The instrument that produces small droplets is called *atomizer*. Atomizers are classified as follows on the mechanisms involved in breaking the bulk of the liquid.

(a) Centrifugal atomizer

e.g. Rotary atomizer. In rotary atomizer the liquid is distributed centrally on a wheel disc or cup that is rotated at high speed. Due to centrifugal force the liquid gets high velocity in the radial direction. The liquid is extended as a thin film over the disc. Then the liquid films disintegrate to produce fine droplets.

Use: Used for slurries or pastes having thermolabile, abrasive, corrosive or high viscous properties.

(b) Pressure atomizer

e.g. Centrifugal pressure nozzle. The liquid is fed through a nozzle under pressure. The feed is issued from the nozzle as a thin film that readily disintegrates into a spray. The feed rotates inside the nozzle so that a cone shaped spray pattern is obtained.

Use: Used to obtain coarse particles.

(c) Pneumatic atomizer

e.g. Two fluid Nozzle atomizer. It involves impact of liquid with a high velocity gas or air. The high velocity gas creates a high frictional force over the liquid surfaces leading to disintegration of liquid into droplets. The stream of gas is rotated inside the nozzle and may come in contact with the liquid wither within the nozzle or after emerging of liquid from the nozzle.

Use: Feed of high viscosity feed produce coarse and less uniform product. Feed of low viscosity produces smaller size particles and more uniform particles.

(B) Spray-Air contact

The spray and hot air can flow in two ways: (i) co-current flow, (ii) in counter-current flow.

In *co-current flow* the spray and the hot air is passed in the same direction. Here fresh droplets come in contact with the hottest air. Solvent evaporates and the droplet temperature falls to wet bulb temperature. So heat labile materials can be dried.

In *counter-current flow* the spray and hot air flows in opposite directions. Here the dried product comes in contact with the hottest air hence this type of flow is suitable for thermostable materials.

(C) Droplet drying

Heat is transferred to droplets by convection from air. The moisture from the interior migrates to the surface and evaporation takes place from the surface. The air temperature is so high that migration of moisture from the interior of the droplet cannot provide the moisture necessary to keep the surface wet. So the surface becomes dried quickly and the moisture in the interior produces vapor that expands the droplets. The final product depends on the nature of the surface layer:

Layer is porous and rigid → No change

Layer is less porous and rigid →Broken particles

Layer is non-porous and plastic→Expanded hollow particles

(D) Separation and recovery of dried products

Majority of dried product falls to the base of the chamber where primary separation takes place. The particles entrained in the air is recovered by any one of the separation equipment like cyclone separator, bag filters, electrostatic precipitators or wet scrubbers.

Applications

1. For drying pharmaceutical products three types of spray dryers are used:

   (i) *Standard open cycle type*: Used from drying infusions, extracts, adrenaline etc. Air is used for drying.
   (ii) *Aseptic open cycle type*: Used to dry antibiotics, vitamins, destrans, hormones, enzymes etc. in aseptic condition. The air is made sterile by passing through HEPA filter.
   (iii) *Closed cycle type*: This type is commonly used to remove organic solvent by using nitrogen instead of air. This type is used for inflammable solvents, for drying toxic substances and for complete recovery of solvent.

2. *For spray congealing*: In this process solid drug particles are suspended in molten waxes (or congealing polymers) and sprayed in a chamber containing cool air. The droplets of wax congeal around the drug particles. Thus the taste of unpalatable drugs can be masked and sustained release products can be produced.

3. *Spray coating*: Solid drug particles are suspended in a coating solution (containing polymer) and the slurry is sprayed in a drying chamber. The solvent of the coating solution evaporates leaving a coating on the solid drug particles. Liquids can also be coated by this method. An emulsion is prepared with the liquid drug as internal phase and coating solution as the external phase. The emulsion is sprayed in the drying chamber. Solvent is evaporated from the external phase leaving a coating over the dispersed liquid drug.

STERILISATION

INTRODUCTION

Among the many facets of patient care is that of the sterilization of a range of materials. These include bacteriologically contaminated glassware and Petri dishes, dressings, sutures, ligatures, surgical instruments, etc., as well as certain raw materials and forms of pharmaceutical dosage. It is considered necessary to sterilise all of these as they could constitute a potential health hazard to patients.

Sterilization is the process of killing or removing microorganisms. A sterile material is one that contains no living organisms at all and the term **sterile** is therefore an absolute one. However, the killing or removing of microorganisms follows a probability function, there always being a finite chance of leaving some live microorganisms intact in the system so that sterilization process are designed to minimize the probability of leaving survivors.

However, with all articles to be sterilized there is the chance that the sterilizing treatment will have a detrimental effect. This is particularly true of pharmaceutical dosage forms where it is important that the chosen process should not cause changes in the formulation, which would reduce its therapeutic efficacy or patient acceptability. For this reason, with the design of all sterilization processes a balance has to be achieved between the maximum acceptable risk of failing to achieve sterility and the maximum permissible concomitant damage caused to the treated articles.

The methods currently used to kill microorganisms involve exposing the preparation to an inimical physical agent or condition for a known period of time. The agents used are of three different types, namely elevated temperature, ionizing or UV radiation, and toxic gases. The fourth way of achieving sterility is restricted to solutions and gases and involves passing the solution through a filter that will retain the microorganisms.

DEFINITIONS

1. **Antiseptic**: A substance that arrests or prevents the growth of microorganisms by inhibiting their activity without necessarily destroying them. Can be applied on human body.
2. **Bactericide:** Anything that kills bacteria.
3. **Bacteriostat:** Anything that arrests or retards the growth of bacteria.
4. **Disinfection:** A process that removes infection potential by destroying microorganisms but not ordinarily bacterial spores. **Disinfectants** are generally meant for application on inanimate objects.
5. **Germicides:** A substance that kills disease microorganisms (i.e. pathogens / germs) but not necessarily bacterial spores.
6. **Sterility:** The absence of viable organisms.
7. **Sterilization:** A process by which all viable forms of microorganisms are removed or destroyed.
8. **Viable =** growing bacteria (or microorganisms) + spores
9. **Vegetative microorganisms =** growing organism

DEFINITION OF STERILISATION

Sterilization is a process whereby all the viable life forms are either killed or removed (by filtration) from a product.

‘Sterilisation’ and ‘Sterility’ are two **absolute** terms i.e. a sterile product should be absolutely free from viable (vegetative + spores) microorganisms.

METHODS OF STERILISATION

STERILISATION METHODS

PHYSICAL

CHEMICAL

Gaseous sterilization

with ethylene oxide

MECHANICAL

Filtration through

bacteria proof

filter units

Dry heat	**Moist heat**	**Radiation**
i)Heating in hot air oven	i) Moist heat in autooclave	i) By exposure to UV - rays
ii)Flaming	ii) Heating with a bactericide	ii) By ionising radiation. (i.e gamma rays)
iii)Infra-red radiation	iii) Heating at 100^0C	
	iv)Pasteurization	
	v)Tyndalization or Fractional sterilisation.	
	vi) Heating at 55 to 60^0C	

i) Berkefeld filter (could be porcelain, keiselguhr)

ii)Sietz filter (Asbestos pads)

iii) Sintered glass filter

iv) Sintered metallic filter

v) Membrane filters

DRY HEAT STERILIZATION

Principle:

The killing of microorganisms by heat is a function of the time-temperature combination used. If the temperature is increased then the time required for killing all the bacteria will be decreased.

Condition: Cycles recommended as per BP 1988 are:

A minimum of 180^{0}C for not less than 30 minutes.

A minimum of 170^{0}C for not less than 1 hour.

A minimum of 160^{0}C for not less than 2 minutes.

Mechanism of killing the bacteria:

The vital constituents of cells such as proteins (enzymes) and nucleic acids are denatured by oxidation.

Biological indicator: Spores of *Bacillus subtilis* var. *niger.* or,

Spores of *Bacillus globigii* or,

Spores of *Clostridium tetani* (atoxigenic)

Application of dry heat sterilization:

Dry heat is used to sterilize

1. **Glass ware** e.g. test tubes, Pasteur-pippettes, petridishes, flasks, glass syringes etc. The glass wares should be prewashed with apyrogenic water.
2. **Porcelain** and **metal** equipment such as forceps, scalpels, scissors etc.
3. **Dry materials** in sealed container like powders.
4. Fats, oils and greasy materials (like petroleum jelly) those are **impermeable** to **moisture**.

Dry heat sterilization is not used in the following cases:

1. **Aqueous solution** cannot be sterilized by dry heat beacause the water will evaporate if the container is kept open. If the container is kept close then it may burst due to pressure developed by the steam.
2. **Surgical dressings** cannot be sterilized by dry heat because cotton or other cellulosic material will get chared at the high temperature.
3. Powders that cannot withstand the high temperature are not sterilised by dry heat method.

HEATING IN A HOT AIR OVEN

- A hot air oven is a double walled chamber made up of mild steel or aluminium. The door is insulated with asbestos gasket to minimize the heat loss. To reduce heat loss by conduction insulating material (glass fibre or puff) is used in between two walls.
- The inner surface is made reflecting to reduce heat loss by radiation.
- It consists of one or more shelves.
- It is thermostatically controlled. Heaters are fitted to heat the air inside. The heat is circulated by normal convection. To spread the heat uniformly forced convection is arranged by fitting fans in strategic places.
- For effective utilization of the oven and to obtain correct results it should be uniformly packed or loaded so that all the objects are exposed to sterilization temperature for the required period of time.
- Care should be taken so that once in operation the oven is not opened in the middle of the cycle.

FLAMING

This is an emergency method, the forceps-tips, the surfaces of the scalpels and the needles may be sterilized by holding the items directly in the flame of a Bunsen burner. This method is generally done in microbiology.

INFRARED RADIATION

- Infrared radiation (IR) is a thermal radiation, i.e. when absorbed by some article its energy is converted to heat and therefore it is often known as radiant energy.
- A tunnel having an IR source is used for this purpose. The instruments and glass wares are kept in trays are passed through this tunnel keeping on the conveyor belt, at a controlled speed exposing them to a temperature of 180^0C for 17 minutes, thereby achieving the sterility. This is a continuous process and is used in hospitals for regular supply of sterile syringes and other apparatus.
- Heating at or above 200^0C by IR in vacuum is employed as a means of sterilizing surgical instruments.
- Cooling is hastened, (after the heating cycle) during the cooling period, by admitting filtered N_2 to the chamber.

MOIST HEAT STERILIZATION

Principle:

Mechanism of killing of microorganisms:

Bacterial death by moist heat is due to denaturation and coagulation of essential protein molecules (enzymes) and cell constituents.

Conditions:

The USP XXI and BP 1988 recommended the following condition:

- Pressure: 15 lb / square inch (psi)
- Temperature: 121^0C
- Time: 15 minutes
- The following combinations of temperature and holding time are normally employed for sterilizing by heat in autoclave:

Holding temperature (^{0}C)	**Holding time (min)**
115 to 118	30
121 to 124	15
126 to 129	10
134 to 138	3

Biological indicator: Spores of *Bacillus stearothermophilus* or

Spores of *Clostridium sporogenes*.

Principles of sterilization by steam under pressure:

Pressure itself has no sterilizing power. Steam is used under pressure as a means of achieving an elevated temperature.

Steam production: This may be achieved in two ways:

1. In a small scale, steam may be generated from water within the sterilizer and since water is present this steam is known as 'wet saturated steam'.
2. For large scale sterilizers dry saturated steam may be piped from a separate boiler.

Penetration of steam:

Steam flows quickly into every article in the load (and into porous articles). This is due to its condensation creating a low-pressure region into which more steam flows.

Rapid heating: The saturated steam heats the load rapidly due to the release of its considerable amount of latent heat.

Moist heat: The condensate produced on cooling hydrates the microorganisms and thus helps in coagulating microbial proteins.

No residual toxicity: The product is free from toxic contamination.

The design and operation of autoclave:

A portable autoclave is an elaborate pressure cooker. It is a hollow cylindrical vessel fitted with a lid that can be tightly secured in a position by nut-bolts or screws. The body is made up of aluminium or steel or gun metal.

The lid is provided with a steam vent, a safety valve and a pressure or temperature gauge. It is heated electrically or gas operated. The electrical element is fitted at the bottom of the autoclave. First water is added so that the heating element is fully immersed in water. Then the materials to be sterilized are placed over a perforated platform.

Heater is switched on. Initially the steam-vent (outlet) is opened. The displaced air is first removed, then the vent is closed. The pressure will then rise to 15 lb/sq inch (psi) and as a result the temperature inside will rise to 121^0C. At this condition the autoclave is kept for 15 minutes, then the heater is switched off. When the pressure inside and outside the autoclave equals, the steam-vent is opened and the lid is removed to take out the sterilized articles.

Application of autoclave:

It is used to sterilize anything, which is not injured by steam and high temperature of sterilization. These includes:

1. Aqueous parenteral solutions e.g. distilled water, saline solutions.
2. Aqueous liquid media e.g. liquid media with or without carbohydrate and gelatin.
3. Surgical dressings and fabrics.
4. Plastic and rubber closures.
5. Metal instruments.
6. Glass apparatus and containers.

HEATING WITH A BACTERICIDE

This method is used for sterilizing aqueous solutions that are thermolabile to withstand normal autoclaving temperature.

Condition:

Temperature: 98 to 100^0C boiling water

Time: 30 minutes

Bactericides: Bactericide that is compatible with the product, container and closure:

e.g. for injection

Chlorocresol	0.2%(w/v)
Phenylmercuric nitrate (PMN)	0.002%(w/v)
Phenylmercuric acetate (PMA)	0.002%(w/v)

e.g. for eye drop

Thiomersal	0.01%(w/v)
Chlorhexidine acetate	0.01%(w/v)
PMA and PMN	0.002%(w/v)
Benzalkonium chloride (BAC)	0.01%(w/v)

Applications:

In the following cases this method can be used:

1. Injection fluids in the final container (i.e. terminal sterilization).
2. Eye drops.

***In the following cases this method cannot be used:*

1. Solution of medicaments intended for intrathecal, intra-atrial, peridural, intra-peritoneal, intra-cisternal and any other route of injection giving access to the cerebro-spinal fluid and for intracardiac and intra-ocularly.
2. Large volume parenteral fluids having a volume greater than 15 ml is not recommended to use bactericide because, though the concentration of the bactericide in the solution is less but the total amount entering into the system may be considerable due to large volume. e.g. bactericides are not used in saline solutions.

TYNDALISATION or FRACTIONAL STERILIZATION

Instrument used : Arnold's sterilizer

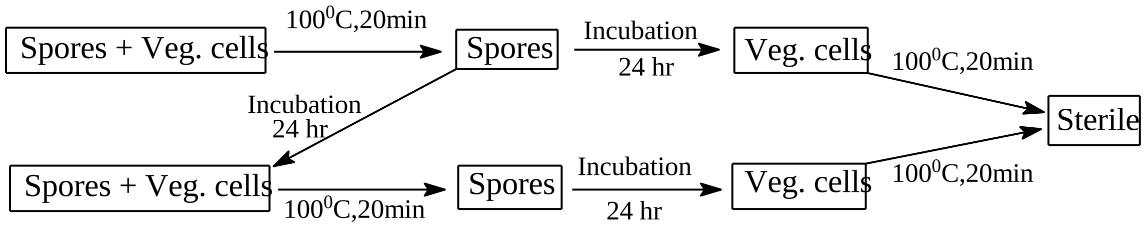
Condition: Arnold's sterilizer employs streaming steam at a temperature of approximately **100^0C** for a period of **20 minutes** for **three consecutive days**.

e.g. Agar media is sterilized by this method.

Principle:

In this process it is presumed that on the first day all the vegetative cells will be killed and some spores may germinate in the following day. So the second day's sterilization cycle will kill the vegetative cells. Similarly some spore may still remain. Therefore for an additional 24 hours period is allowed to elapse to make sure all ‘spores’ have germinated into vegetative cells.

N.B.



Causes of failure in this process:

It may be seen unless the spores are germinated the method will fail to sterilize. Failure may be due to the following reasons:-

1. The media may be unsuitable for the germination of the spores, e.g. distilled water is not a favorable environment for the growth of bacteria and will not permit spores to germinate into vegetative cells.
2. Spores of anaerobic bacteria, if present, will not germinate in a media in contact with air.
3. If any preservatives are present in the product it may not allow to germinate the spores.
4. If a product has intrinsic antimicrobial properties then it will not allow to spore germination.

Application of Arnold’s sterilizer (Tyndallization)

The Arnold’s sterilizer is used principally for the sterilization of gelatin, milk and carbohydrate media. Higher temperature in the autoclave, larger single exposure in the Arnold’s sterilizer may hydrolyze or decompose carbohydrates and prevent gelatin from solidifying. Obviously such media would then be unsuitable for use.

PASTEURIZATION

Pasteur developed the procedure of gentle heating (Pasteurization) to prevent spoilage of beers, wines by undesired contaminating microbes. This process has no pharmaceutical application. This is used for processing of milk in order to kill the pathogenic bacteria without affecting the taste and nutritional value of the milk. Following two methods are used:

Holding method:

The milk is heated in tanks at **62^0C for 30 mins** while it is gently stirred and the steam is flown on the surface to disperse the foam.

High temperature short time method (H.T.S.T. / **Flash method**):

In this process the milk is rapidly raised to **71.6^0C** held at this temperature for at least **20 seconds** and quickly cooled.

The common milk born pathogens (*Mycobacterium tuberculosis, M. bovis, Brucella abortes*) do not form spores and are reliably sterilized by this procedure. In addition to this, total bacterial count is generally reduced by 97 - 99%. therefore in true *sense this technique is not an absolute sterilization process*.

Advantages of moist heat sterilization:

1. High heat content plus rapid heat transfer.
2. Destroys micro-organism more efficiently than dry heat.
3. It can be used for a large number of injections, ophthalmic solutions, irrigants, dialysis fluids etc.
4. It rapidly penetrates porous materials and is therefore very suitable for sterilizing surgical dressings and materials.
5. The process is adaptable for plastic containers and some other special dosage forms.
6. It is more suitable than dry heat for sharp instruments.
7. Accurate control and monitoring of the process is possible.
8. No toxic contaminants are left in the materials sterilized.

Disadvantages of moist heat sterilization:

1. It is not suitable for anhydrous materials such as powders and oils.
2. It cannot be used for thermolabile substances.
3. It does not destroy pyrogens.

GASEOUS STERILIZATION

This process involves exposure of materials to sterilize gasses such as *ethylene oxide, formaldehyde, glutaraldehyde, propylene oxide*.

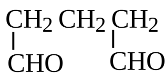
ETHYLENE OXIDE

Ethylene oxide is the only gas that is successfully used on a large scale of industrial and medical applications.

Structure:

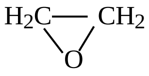
HCHO

Formaldehyde

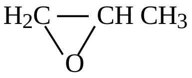


$\underset{CHO}{\underset{|}{CH_2}}\,CH_2\,\underset{CHO}{\underset{|}{CH_2}}$

Glutaraldehyde



Ethylene oxide



Propylene oxide

Mechanism of action: Ethylene oxide acts by alkylation.

Biological indicator: *Bacillus subtilis* var. *niger*.

Factors affecting sterilization:

Sterilization efficiency is influenced by

1. the concentration of ethylene-oxide
2. the humidity of the sterilizing atmosphere
3. the temperature of sterilization
4. time of exposure
5. physical nature and permeability of the load.
6. atmospheric preconditioning of the load before sterilization.

FORMALDEHYDE

Like ethylene oxide this is an alkylating agent but it is generally inferior for use as a sterilizing agent. Because

- formaldehyde has poor penetrating power and is readily inactivated by organic matter.
- High concentrations are difficult to maintain in the atmosphere because it tends to deposit in the form of solid polymers on contact with cool surfaces.

Condition:

Input concentration: 2 g/L of HCHO in subatmospheric steam

Temperature: 90^0C

Exposure time: 3 hours

Formaldehyde can be obtained as :

1. Formaldehyde solution (Formalin) B.P.

   Approximately 37%w/w containing stabilizers to prevent deposition of solid polymers

2. Tablets of paraformaldehyde

* In case of formalin addition of $KMnO_4$ produces heat by oxidation and the gas will be vaporized.

Use: Dressing packs.

Disadvantages:

1. Low penetrability. It cannot penetrate polymeric packaging.
2. Polymerize to inactive forms on the surface of low exposure temperature.
3. Can fall in the active concentration
4. Cannot sterilize narrow lumen.

<u>Solution of the above problems:</u>

1. Reducing the size of the load.
2. Increasing exposure temperature.

STERILIZATION BY RADIATION

Radiation can be divided into two groups:

1. Electromagnetic waves: (i) infra-red radiation (IR) (iii) X-rays
(ii) ultraviolet radiation (UV) (iv) gamma rays

2. Streams of particulate matter (i) alpha ra

For sterilization infrared, ultraviolet, gamma radiation and high velocity electrons (a type of beta radiation) are used for sterilization.

Radiation	**Wave length**	**Energy**
UV-radiation	190 to 370 nm	5 eV
Gamma radiation	1 to 10^{-4} nm	1.3 MeV*
High velocity electrons		4MeV

* MeV = Million electron volt

ULTRAVIOLET RADIATION

Source: Low current of high voltage is passed through mercury vapor in an evacuated tube made of borosilicate glass.

Dose of sterilizing radiation: 10 to 60 microwatts / cm^2 reduce the populations of vegetative cells by 90% in a short period.

Mode of action: Only a narrow range of wavelength (220 to 280 nm) is effective in killing micro-organisms, and wavelengths close to 253.7 nm are the most effective. It has been found that a maximum biological efficiency exists at 253.7 nm, which is also the absorption peak of isolated DNA, this suggests strongly therefore that DNA or the nucleic acid is the target for UV-induced lethal events.

Use: (i) Surface sterilization

(ii) Sterilization of clean air and water in thin layers.

Disadvantages:

1. It has very poor penetration power.

   [N.B. UV radiation does not penetrate normal packaging material, such as glass or plastic and hence, it is not used to sterilize pharmaceutical dosage forms.]

2. UV-light sterilization is not absolutely reliable because DNA may get repaired in some favorable condition.

3. Bactericidal UV -light causes eye problems and erythema (reddening) of the skin if those parts are exposed to UV- radiation for a prolonged period of time.

IONISING RADIATION

Mode of action of ionizing radiation:

Ionizing radiation can cause excitations, ionization and where water is present free radical formation. Free radicals are powerful oxidizing (OH, HO_2) and reducing (H) agents, which are capable of damaging essential molecules (enzymes and DNAs) in living cells. This results in cell death.

Biological indicator: *Bacillus pumilis.*

HIGH SPEED RADIATION

Source: This type of sterilizing radiation is most widely used in Denmark and the USA. In a machine known as a van de Graph accelerator electrons are generated from a suitable source and then accelerated along a highly evacuated tube by a tremendous potential difference between the ends.

Dose: 5 MeV to 10 MeV (Million electron Volt).

Use: The beam, which is narrow and intense, is used to irradiate articles on a conveyor-belt.

GAMMA RAYS

Source: Radiation from the radioactive isotope of Cobalt ^{60} Co, is used as a source of gamma emission.

Dose: 1.25 MeV,

Some users take the adequate dose as 2.5 Mrad (Mega radiation unit)

Procedure: Articles for sterilization by radiation are packed in boxes of standard size, which are suspended from a monorail and sterilized by slow-passage around the gamma-ray source.

Uses:

- Articles regularly sterilized on a commercial scale include plastic syringes, catheters, hypodermic needles and scalpel blades, adhesive dressings, single-application capsules of eye-ointment and catgut
- Containers made of polyethylene and packaging materials using aluminum foil and plastic films.

Disadvantages:

- The damage of the cells is mediated through radiation-induced free radicals in water, hence the extent of degradation is found to be maximum in pharmaceuticals in aqueous solutions. Practically the amount of degradation in aqueous solution is so great that this sterilization method is only confined to sterilize surgical sutures, instruments etc.
- The necessary apparatus is much expensive for installation in hospital. It is employed commercially for the sterilization of large amount prepackaged disposable items such as plastic syringes and catheters, which are unable to withstand heat.

STERILIZATION BY FILTRATION

This method is used for sterilizing thermolabile solutions, which will otherwise be degraded by other conventional heating methods.

The drug solutions are passed through the sterile bacteria proof filter unit and subsequently transferring the product aseptically into the sterile containers which are then sealed.

The process involves considerable hazards. Hence IP and BP require that the tests for sterility be carried out on the filtered product.

Biological indicator: *Micromonospora diminuta*.

Procedure:

The solutions to be sterilized is passed through the filter and collected in the sterile receiver by the application of positive pressure to the nonsterile compartment or negative pressure to the sterile side..]

Advantages of sterilization by filtration:

1. Thermolabile solutions can be sterilized.
2. It removes all the living microorganisms.

Disadvantages of sterilization by filtration:

1. Filters may break down suddenly or gradually on use.
2. Sterility testing is obligatory on the filtered solution.
3. Filter media may be absorbed on the filter surface.
4. Viruses are not removed by filtration.
5. Suspensions and oils cannot be sterilized by this method due to their heavy load of particulate matters and viscosity.

Mode of action:

The filters are thought to function by one or usually a combination of the following:

1. Sieving or screening,
2. Entrapment,
3. Electrostatic attraction.

When a particle is larger than the pore size of the filter the particle is retained on the filter - this known as *sieving or screening*.

Entrapment occurs when a particle smaller than the size of the pore enters into the pore channel and lodges onto the curves of the channel while passing through it.

Electrostatic attraction causes particles, opposite in charge to that of the surface of the filter pore, to be held or adsorbed onto the surface.

MEMBRANE FILTERS

- Membrane filters are made of cellulose-derivative (acetate or nitrate). They are very fine. They are fixed in some suitable holders.
- **Nominal pore size is 0. 22 ± 0. 02 μm** or less is required.
- The membranes are brittle when dry. In this condition they can be stored for years together. They become very tough when dipped in water.

- They are sterilized by autoclaving or by ethylene oxide gas. They cannot be sterilized by dry heat as they decompose above 120^0 C.
- They are suitable for sterilizing aqueous and oily solutions but not for organic solvents such as alcohol, chloroform etc.
- Membrane filters are generally blocked by dirt particles and organisms. Pre-filtration (through glass-fibre paper prefilter) reduces the risks of blockage of the final filter.

Examples of membrane filters:

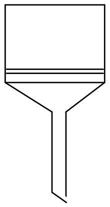
MF-Millipore – it is a mixture of cellulose esters

Sartorius Regular – it is made of cellulose nitrate

Gelmen Triacetate Metricel – cellulose triacetate

SINTERED (or FRITTED) GLASS FILTERS

Borosilicate glass is finely powdered in a ball-mill and the particles of required size are separated. This is packed into disc mounted and heated till the particles get fused. The disc thus made have pore size of 2 μm and are used for filtration.



They are cleaned with the help of sulfuric acid.

SIETZ FILTER: They are made of asbestos pad.

[For further details see Cooper & Gunn Dispensing pp. 582]

CERAMIC FILTERS: They are made of either porcelain or keiselghur. These are supplied in the shape of candles mounted to metallic joint.

TESTING OF FILTERS:

The BP requires that the integrity of an assembled sterilizing filter be verified before use and confirmed after use by means of a suitable test.

Bacteriological test:

A diluted solution of broth culture of *Serratia marcescens* is passed though the filter and the filtrate is collected aseptically and incubated at 25^0C for 5 days. The filter passes this test if no growth is found after 5 days of incubation.

Bubble point test:

The bubble point of a test filter is the **pressure** at which the largest pore of a wetted filter is able to pass air.

Objectives:

1. Filtration should normally be performed at pressures lower than the bubble point of a membrane. This prevents gas from passing through the filter at the end of a filtration cycle and thereby **prevents excessive foaming**.

2. For testing membrane efficiency and integrity.

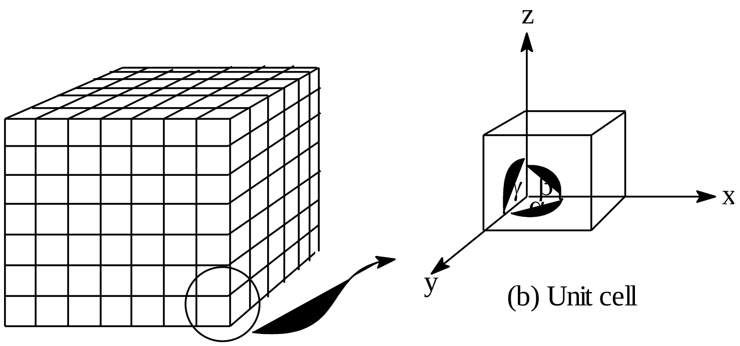
INTRODUCTION

Crystallization

Crystallization is the formation of solid particles within a homogeneous phase. It may occur as the formation of solid particles in a vapor, as in snow; as solidification from a liquid melt, as in the manufacture of large single crystals; or as crystallization form liquid solution.

Crystal

A **crystal** is a regular polyhedral form, bounded by smooth faces, which is assumed by a chemical compound, due to the action of its interatomic forces, when passing, under suitable conditions, from the state of a liquid or gas to that of a solid.



(a) Lattice structure in crystal

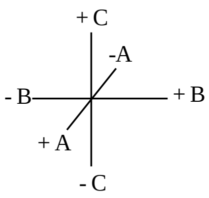
(b) Unit cell

[N.B. A polyhedral form simply means a solid bounded by flat planes (we call these flat planes **CRYSTAL FACES**). "A chemical compound" tells us that all drugs are chemicals. The last half of the definition tells us that a crystal normally forms during the change of matter from liquid or gas to the solid state. In the liquid and gaseous state of any compound, the atomic forces that bind the mass together in the solid state are not present. Therefore, we must first crystallize the compound before we can study it's geometry. Liquids and gases take on the shape of their container, solids take on one of several regular geometric forms. These forms may be subdivided, using geometry, into six systems.]

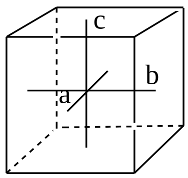
Crystal Lattice

Crystal lattice is defined as a three dimensional network of imaginary lines connecting the atoms, ions or molecules.

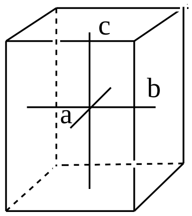
The distance between the center of two atoms (or ions, or molecules) is called *length of unit cell* and the angle between the edges of a unit cell is called as *lattice angle*.



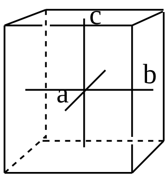
Crystallographic axes



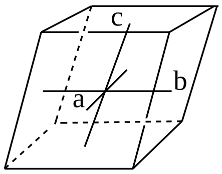
Cubic
$a = b = c$
$\angle = 90^0$



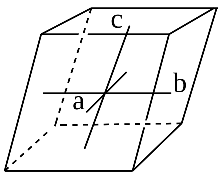
Tetragonal
$a = b \neq c$
$\angle = 90^0$



Orthorhombic
$a \neq b \neq c$
$\angle = 90^0$



Monoclinic
$a \neq b \neq c$
$\angle bc \neq 90^0 \quad \angle ac = \angle ab = 90^0$



Triclinic
$a \neq b \neq c$
$\angle bc \neq \angle ac \neq \angle ab \neq 90^0$

- The units that constitute the crystal structure are atoms, ions or molecules.
- Ions with opposite charges are bonded together by electrostatic attraction e.g. Na^+ and Cl^- ions bonded in NaCl crystals.
- Atoms are bonded together by covalent bonds e.g. C atoms are bonded by covalent bonds in diamond and graphite.
- Molecules of organic compounds are bonded by van der Waal's force and / or hydrogen forces. E.g. Naphthalene, p-hydroxy benzoic acid.

Crystal Forms

Crystal lattice can be classified according to the angles between the faces. There is only finite number of symmetrical arrangements possible for a crystal lattice, this is termed as crystal forms.

The ability of a compound to exist in different crystal forms is known as **polymorphism**.

[N.B. The types of crystal-forms has no relationship to the relative sizes of the faces since the relative development of the faces are not constant, only the angles between the faces remain constant.]

There are six types of crystal forms, depending on the arrangement of the faces expressed as *crystal axes* and *angles between the axes*.

1. **Cubic** - The three crystallographic axes are all equal in length and intersect at right angles (90 degrees) to each other. [a = b = c] e.g. Sodium chloride and potassium chloride crystals.
2. **Tetragonal** - Three axes, all at right angles, two of which are equal in length (a and b) and one (c) which is different in length (shorter or longer). Note: If c was equal in length to a or b, then we would be in the cubic system. Urea and potassium dihydrogen phosphate crystals.

3. **Orthorhombic** - Three axes, all at right angles, and all three of different lengths. Note: If any axis was of equal length to any other, then we would be in the tetragonal system e.g. Barium sulphate crystals.
4. **Hexagonal** - Four axes, three of the axes fall in the same plane and at 60^0 to each other. These 3 axes, labeled a1, a2, and a3, are the same length. The fourth axis, termed c, may be longer or shorter than the ‘a’ axes set. The c axis also passes through the intersection of the a axes set at right angle to the plane formed by the a set. Ice and thymol crystals.
5. **Monoclinic** - Three axes, all unequal in length, two of which (a and c) intersect at an oblique angle (not 90 degrees), the third axis (b) is perpendicular to the other two axes. Note: If a and c crossed at 90 degrees, then we would be in the orthorhombic system. Sucrose and Ephedrine chloride crystals.
6. **Triclinic** - The three axes are all unequal in length and intersect at three different angles (any angle but 90 degrees). Note: If any two axes crossed at 90 degrees, then we would be describing a monoclinic crystal. Phenolphthalein and Copper sulphate.

Crystal Habits

Crystal is a polyhedral solid with number of planar faces. The arrangement of these faces is termed as habit. The crystal habit may change due to changes in rate of deposition, shielding of certain faces, presence of impurities in mother liquor.

e.g. NaCl crystallizes out from aqueous solution with cubic faces only. On the other hand, if NaCl is crystallized from aqueous solution containing a small amount of urea, the crystals are found to have octahedral faces.

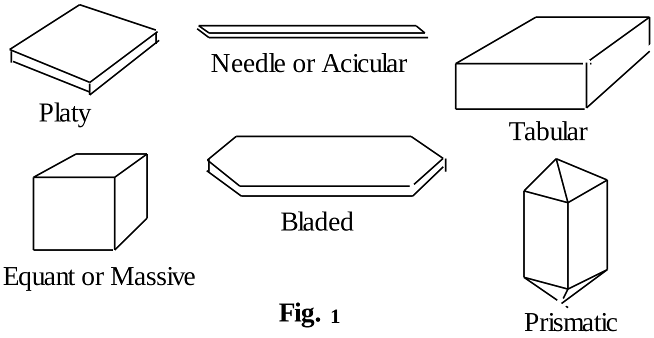


Fig. 1

Different crystal habits are

Acicular e.g. Nalidixic acid

Columnar e.g. Fluorocortisone acetate

Blade e.g. Resorcinol

Plate e.g. Naphthalene

Tabular e.g. Tolbutamide

Equant e.g. Sodium chloride etc.

Purity of crystals

A well formed crystal is nearly a pure chemical. Some times it retains mother liquor when removed from the final magma. If the product contains crystal aggregates then considerable amount of impure mother liquor may be entrapped inside the product. When this mother liquor is dried on the crystals, contamination results.

In practice most of the mother liquor is removed from the crystals by centrifuging or filtration and the balance is removed by washing with fresh solvent.

IMPORTANCE OF CRYSTALLIZATION

- Crystallization from solution is important industrially because of the variety of materials that are marketed in the crystalline form.
- Crystallization affords a practical method of obtaining pure chemical substances in a satisfactory condition for packaging and storing. A crystal formed from an impure solution is itself pure (unless mixed crystals occur).
- A drug may remain in different crystalline forms, some are stable, and rests are metastable. The metastable forms have greater solubility in water, thus have better bioavailability. By controlling the conditions during crystallization the quantity of metastable to stable forms may be controlled.
- After crystallization water or solvent molecules may be entrapped within the crystal structure and thus form hydrates or solvates which have different physical properties that may be utilized in various pharmaceutical purpose.
- Particles with various micromeritic properties, compressibility and wettability can be prepared by controlling the crystallization process.
- Desalination of seawater by crystallization method requires only 1/7th of the energy required by distillation process.

Factors affecting the crystal habit

1. *Presence of another substance in the mother liquor*:

   Sodium chloride crystallized from aqueous solutions produces *cubic* crystals. If sodium chloride is crystallized from a solution containing a small amount of urea, the crystals obtained will have octahedral faces. Both types of crystals belong to the cubic crystal form but differ in habit.

2. *Solvent:*

   Griseofulvin crystallized out from acetone has different crystal habit than when crystallized from benzene or chloroform.

3. *Rate of cooling*:

   Acicular or needle-like crystals are produced when the solution is cooled very slowly. Fluffy and small crystals are produced when the solution is cooled very fast.

THEORY OF CRYSTALLIZATION

Mechanism of crystallization

The formation of crystals from solution involves three steps:

A. Supersaturation
B. Nucleation (or nucleus formation)
C. Crystal growth

A. Supersaturation

When the concentration of a compound in its solution is greater than the saturation solubility of that compound in that solvent the condition is known as *supersaturation*. This is an unstable state. From this supersaturates solution the escess compound may be precipitated out or crystallize.

Supersaturation can be achieved by the following methods:

1. Evaporation of solvent from the solution.
2. Cooling of the solution.
3. Formation of new solute molecule as a result of chemical reaction *in situ*
4. Addition of a substance, which is more soluble in solvent than the solid to be crystallized.

B. Nucleation

Nucleation refers to the birth of very small bodies of molecules from which the crystal forms.

- In solution, solute molecules, ions or atoms remain in constant random motion. This is due thermodynamic energy of the solution system.
- When the solute particles (molecules, atoms or ions) moves and collide over each other they may form aggregates. This aggregates are called *clusters*. These are loose aggregates, which usually disappear quickly.
- Some clusters may become so big that they may arrange themselves in lattice arrangement. These bodies of aggregates are called *embryo*. However, embryos are unstable and they may break into clusters again.
- Some embryo may grow to such a size that it remains in thermodynamic equilibrium with the solution. They do not revert back to clusters. These bodies are called *nucleus* (plural is *nuclei*).

C. Crystal growth

Crystal growth is a diffusion process and a surface phenomenon. Every crystal is surrounded by a layer of liquid known as *stagnant layer*. From the bulk solution a solute particle (molecule, atom or ion) diffuse through this stagnant layer and then reaches the surface of the crystal. This particles then organize themselves in the crystal lattice. This phenomenon continues at the surface at a slow rate. This process will happen if the bulk solution is supersaturated.

Mier’s Supersaturation theory

Mier and Issac proposed a theory explaining a relationship between supersaturation and spontaneous crystalization.

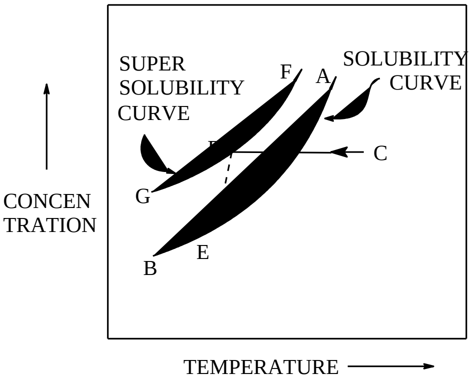
Mier’s theory points out that

(i) the greater the degree of supersaturation, the more chance is of nuclei formation,
(ii) if the super-saturation passes a certain range of values, nuclei formation is extremely rapid.

Assumption:

1. The solute and the solvent must be pure.
2. The solution must be free from solid solute particles.
3. The solution must be free from foreign solid particles.

The theory can be explained with the help of *solubilty - supersolubility* diagram.



- Here the curve AB is the ordinary *solubility* (equilibrium) *curve*. It represents the maximum concentration of solutions that can be obtained by bringing solid solute into equilibrium with solvent.
- If a sample of solution having a temperature and composition of point C is cooled in the direction of CD, it first crosses the solubility curve AB, but no nucleus will be formed. When it reaches some where in the neighbourhood of the point D (according to Mier’s theory) crystallization begins. As the crystallization proceeds the concentration of the solution follows roughly according to the curve DE and reaches the solubility curve.
- In the absence of any solid particles the curve FG represents the limit at which nucleus formation begins spontaneously and, consequently crystallization starts – this line (FG) is called the *super-solubility curve*. According to Mier’s theory at any point between C and D points nuclei cannot form and crystallization cannot start.

Limitations of the Mier’s theory

1. According to Mier’s theory, crystallization starts at super-solubility curve (FG). But the general tendency is that crystallization takes place in an area rather than a line.
2. If the solution is kept for long periods, nucleation starts well below the super-solubility curve.
3. If the solution is available in large volume, nucleation starts well below the super-solubility curve.
4. Mier’s theory is applicable only when pure solute and pure solvent is taken. In practice, it is impossible to get them in pure state.

[N.B. Mier’s theory is base on the postulation that the solution consists of pure solvent and pure solute without the presence of any solid particles, whether of solute itself or of any foreign material. In presence of any such solid particles it has been found that crystallization occurs well before the line FG.]

5. During crystallization the solution may become contaminated with dust, particles from container etc. Nucleation may be initiated from these foreign particles also.

Solubility curves

Solubility of a solute depends on the temperature. When the solubility (of a saturated solution) of a solute is plotted against temperature the curve is known as *solubility curve*. Temperature is plotted in X-axis and solubility is plotted in Y-axis. The metastable condition is shown by dotted line.

The following solubility curves may be observed with various solutes:

1. *Curve-1* represents potassium nitrate (KNO_3). This is most common type in which the solubility of a substance increases with temperature.
2. *Curve-2* represents sodium chloride (NaCl). The solubility increases with increase in temperature, but to a marginal extent.
3. *Curve-3* represents sodium thiosulphate ($Na_2S_2O_3$). Here solubility increases rapidly with temperature. But inflections are observed in the curve to represent different hydrates.
4. *Curve-4* represents sodium carbonate (Na_2CO_3). This curve is unusual. Here solubility of sodium chloride increases with temperature, if it is in hydrated form.. Once the compound turns in to mono-hydrate form, its solubility decreases.

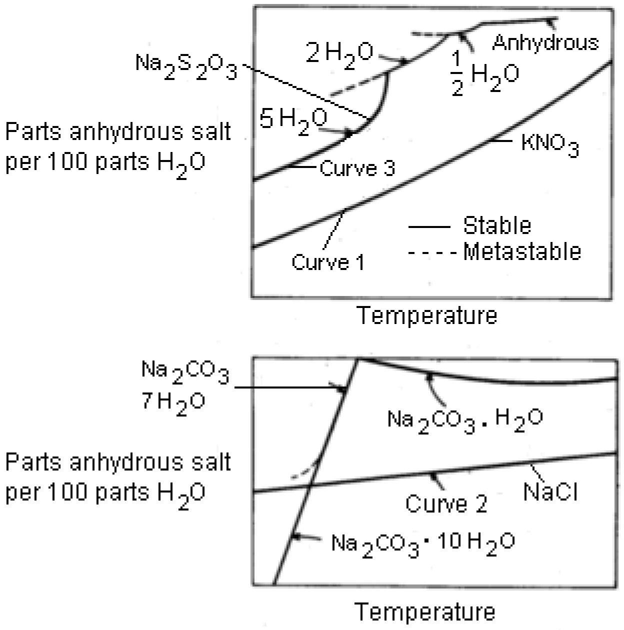


Fig. Typical solubility curves of certain substances

Calculation of yields

A solution of a substance is taken. The concentration of the solute in the solution is noted.

The solution is evaporated or cooled to make it supersaturated solution. The excess solute crystallizes out of the solution and the mother liquor at the end of the crystallization process is nothing but a saturated solution. The solubility is the *saturation solubility* at that temperature. This saturation solubility at that temperature is determined from the solubility curve.

During evaporation some amount of solvent is evaporated.

Material may crystallize out in pure form or as hydrates.

Problem:

A solution containing 30% $MgSO_4$ *and 70%* H_2O *is cooled to* 18^0*C. During cooling 5% of the total water in the system evaporate. How many kilograms of crystals are obtained per kg of original mixture? Crystals formed are* $MgSO_4$*,* $7H_2O$*. Concentration of mother liquor is 24.5% anhydrous* $MgSO_4$*.*

Solution

In the original mixture

MgSO4 30% x 1000kg = 300kg

Free water 70% x 1000kg = 700kg

Total = 1000kg

Water evaporated = 5% of free water in the original mixture = 5% x 700kg = 35kg

$MgSO_4$ crystallizes out in the form of $MgSO_4$, $7H_2O$ (7 molecules of crystal water)

Let, **m** kg of ($MgSO_4$, $7H_2O$) crystal forms from 1000kg of original mixture.

Molecular weight of MgSO4 = 120.4 and $MgSO_4$, $7H_2O$ = 246.5

Therefore, **m** kg $MgSO_4$, $7H_2O$ crystal contains $\mathrm{m}\, x \frac{120.4}{246.5}$ kg of $MgSO_4$. = 0.488 m kg

$MgSO_4$.

and $\mathrm{m}\, x \frac{7x18}{246.5}$ kg water = 0.511 m kg H_2O

In the final mixture

$MgSO_4$, $7H_2O$ crystal = m kg

$MgSO_4$ left in the mother liquor = (300 – 0.488m) kg

Water left in the mother liquor = (700 – 35 – 0.511m) kg = (655 – 0.511m) kg

From the solubility curve of $MgSO_4$ in water it is found that at 18^0C the solubility of $MgSO_4$ is 24.5%.

So in the final mixture at 60^0F $\quad MgSO^4 \quad = \quad 24.5$kg

Free water = (100 – 24.5) kg = 75.5 kg

Therefore, $$\frac{300 - 0.488m}{655 - 0.511m} = \frac{24.5}{75.5}$$ Solving this equation will yield, m = 261 kg

CRYSTALLIZERS

Classification of crystallizers

Crystallization equipment is classified by the methods by which supersaturation is bought about. These are as follows:

1. Supersaturation by cooling alone
    - A. Batch processes
        - (i) Tank crystallizers
        - (ii) Agitated batch crystallizers
    - B. Continuous processes
        - (i) Swenson-Walker
        - (ii) Other
2. Supersaturation by adiabatic cooling
    - A. Vacuum crystallizers
3. Supersaturation by evaporation
    - A. Salting evaporators
    - B. Krystal evaporators

TANK CRYSTALLIZER

Procedure

Hot , nearly saturated solutions are kept in open rectangular tanks in which the solution stood while it cooled and crystals are deposited. No seed is given. Some times rod or strings are hung in the tanks to give the crystals additional surfaces on which the

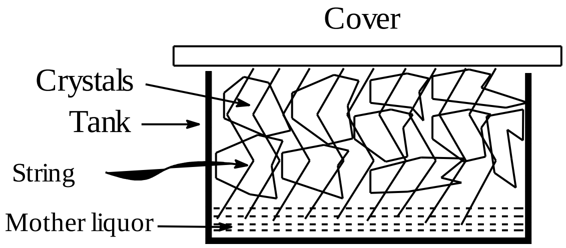


Fig. Tank crystallizer

crystals may grow and to keep major part of the product above the bottom of the tank where the sediment is collected (actually the sediment is the source of impurity).

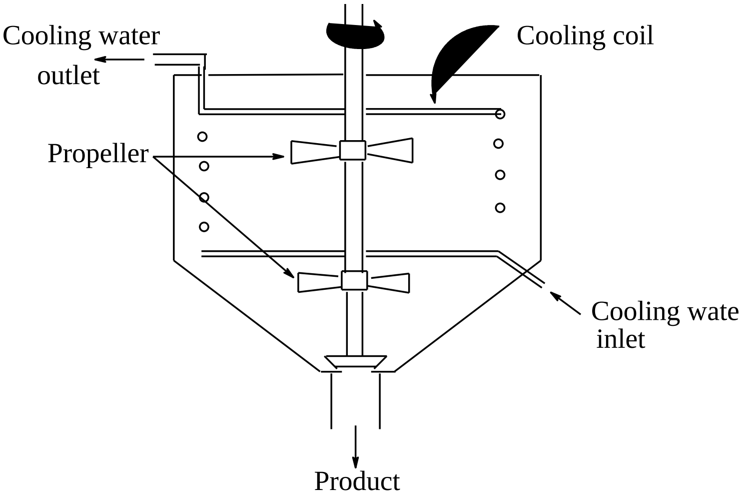
Disadvantages

1. Crystal growth is very slow.
2. Crystals formed are large and interlocked, so mother liquor along with impurity gets entrapped within the crystals.
3. The floor space required and the amount of material tied up in this process are both large.

AGITATED BATCH CRYSTALLIZER

Procedure

It is a tank with a central shaft running through it. Water is circulated through the cooling coils, and the solution is agitated by the propellers on the central shaft. Product is collected at the bottom of the crystallizer. It is a batch process.



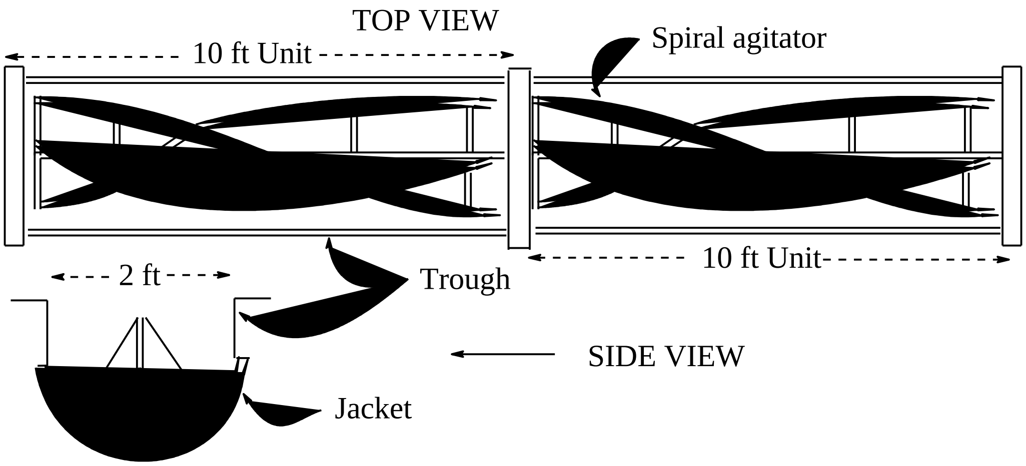
Advantages

- The agitation increases the rate of heat transfer and keeps the temperature of the solution uniform through out the crystallizer.
- Agitation keeps the smaller crystals in suspension and allows them to grow uniformly– thus finer crystals can be obtained.

Disadvantages

- It is a batch process or a discontinuous one.
- Since the solubility is least at the cooling surface hence the crystals growth is more rapid on the cooling coils. Thus the crystals deposited on the cooling coils reduces the heat transfer rate.

SWENSON-WALKER CRYSTALLIZER



Construction: It consists of an open <u>trough</u> (A) 2 ft wide, with a semicylindrical bottom. A water <u>jacket</u> (B) is welded to the outside surface of the trough. Inside the trough a slow speed, long pitch, spiral <u>agitator</u> (C) is fitted as close as possible to the bottom of the trough. The agitator rotates at a speed of 7 rpm.

This apparatus is built in units of 10 ft length. Several such units are joined together to give increased capacity.

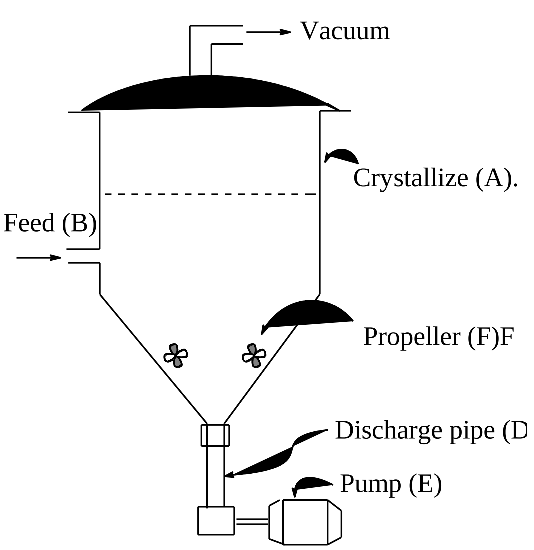
Working principle: This is continuous type crystallizer. The hot supersaturated solution is fed at one end of the trough, and the cooling water is flows in the jacket, but in counter current (i.e. opposite to the flow) to the solution. As the hot solution flows along the trough it is cooled and crystals are formed. Agitator prevents an accumulation of the crystals on the cooling surface and, lift the crystals and shower them through the solution. In this manner perfectly individual crystals are formed.

At the end of the crystallizer there may be an over flow gate where the mother liquor and the crystals are overflowed in a draining table or drain box,, from which the mother liquor is separated and fed in the crystallizer again. The crystals are sent to centrifuge.

In another method an inclined screw conveyor lifts the crystals and the wet crystals are send to the centrifuge.

VACUUM CRYSTALLIZER

***Principle**:* Under vacuum the boiling point of a liquid reduces. So under vacuum a liquid boils under its normal boiling point. If a warm saturated solution is introduced into a vessel in which a vacuum is maintained and the feed temperature is above the (reduced) boiling point of the solution then the solution so introduced must flash (sudden evaporation) and be cooled due to adiabatic evaporation (taking the latent heat from the solution). Cooling will cause supersaturation and



thus crystallization. Evaporation will increase the yield.

Vacuum crystallizers are often operated continuously, but they can also be operated batch-wise.

Construction

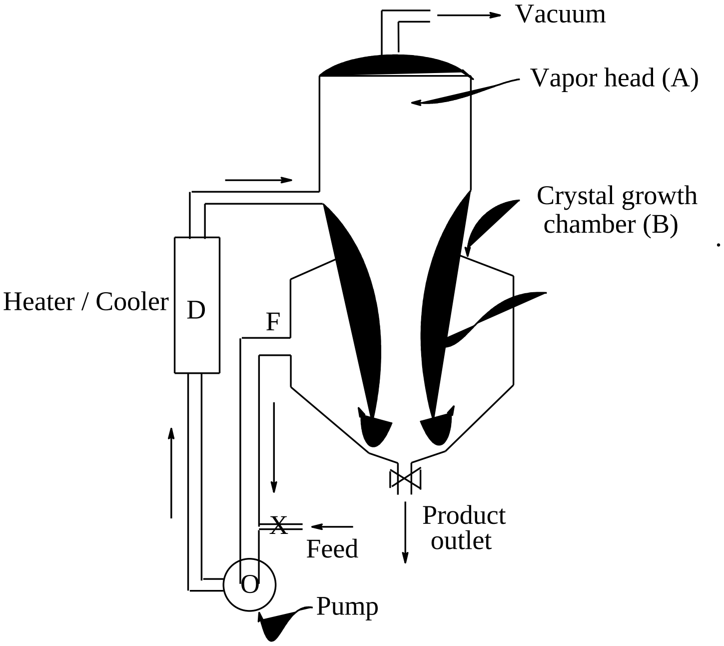
A simple vacuum crystallizer contains no moving parts. The crystallizer is a cone-bottomed vessel (A). The feed enters at any suitable point (B) of the crystallizer and the vapor leaves at point C to go to the vacuum producing equipment. Under vacuum the feed flashes (rapid evaporation) and due to ebullition (formation of bubbles) in the crystallizer the crystals are kept in suspension until they become large enough to fall into the discharge pipe (D), from which they are removed as slurry by a pump (E).

There is sometimes a tendency for the feed to short-circuit to the discharge pipe without being flashed (i.e. the feed enters and directly flows into the discharge pipe). For this reason two propellers (F) are installed in the crystallizer to keep the solution thoroughly stirred to prevent the feed solution from reaching the discharge pipe without flashing.

KRYSTAL CRYSTALLIZER

Construction and working principle

Here A is the *vapor head*, and B is the *crystallizing chamber*. For the first time solution is fed into the suction end of the pump (C). Pump sends the feed solution to the heater or cooler (D). The feed then is introduced in the vapor head (A). The vapor is discharged to a condenser and vacuum pump. The operation is so controlled that the crystals are not formed in the vessel A, but the vessel A is prolonged into tube E extended almost to the bottom of vessel B. At the lower part of the vessel B the crystals are formed and are suspended in the liquid. The supersaturated liquid formed in nozzle E passes to vessel B and an upward flow maintains the suspension at the bottom of vessel B.



At the bottom coarser crystals remain and becomes finer at the top. The coarser crystals are drawn out form time to time through G. The finest crystals, remaining at the top flows again through connection F to the pump which is sent again into the heater or cooled D.

Use:

Krystal crystallizer is preferred when large quantities of crystals of controlled size is required. For example in sodium chloride and magnesium sulphate crystallization.

CAKING OF CRYSTALS

What is caking?

Caking can be defined as the process of formation of clumps or cakes when crystals are improperly stored.

Crystal powders can absorbs moisture when the humidity of the air is above the critical humidity. Below this critical humidity the crystals do not absorbs moisture while above the critical humidity the crystals absorbs moisture and forms a saturated solution on the surface of the crystals. When temperature of the crystals are cooled (due to some reason) or the water is evaporated from the surface, the extra solute crystallize out and thus may form solid bridges in between two adjacent crystals, called *crystal bridge*s. Thus the crystal particles will join together to form hard aggregate. This aggregates or lumps are called caking.

Problems of caking

After caking the flow properties of the powder decreases. Powder will not flow uniformly from the hopper into the die-cavity of tablet punch machine. Capsule filling will not be uniform. Filling of pouches will not be uniform.

Factors affecting caking

1. *Size of the crystals*: Smaller crystals have a greater tendency of caking than larger crystals because powder of smaller crystals have less void so greater number of contact points. More the number of contact points greater number of crystal bridges will be formed.
2. *Shape of the crystals*: Spherical shape posses the least possible points of contacts than any other form. Hence, the distorted crystals tends to more caking than spherical particles.
3. *Humidity*: The higher the humidity of the atmosphere to which crystals are exposed, more will be the rate of caking.
4. *Time of exposure to moisture*: The higher the time of exposure, the more will be the caking, provided that the atmosphere has humidity more than critical humidity.
5. *Impurities in the crystals*: The crystals may be coated with impurities from the mother liquor. This may increase the value of critical humidity. For example $MgCl_2$ and $CaCl_2$ alters the critical humidity of NaCl crystals.
6. *Melting points of crystals*: If the melting point of the crystals are near room temperature then at slightly high temperature they will melt and at low temperature they will fuse to form crystals and thus increases caking.
7. *Temperature fluctuations*: When temperature is increased solubility of crystals increases. Subsequent decrease of temperature will produce a supersaturated solution from which crystals will be precipitated. So fluctuations of temperature produce crystals rapidly.

Prevention of caking

1. Crystals must be more spherical in shape, with least points of contact.
2. Crystals must be larger in size with more voids ad must be of a narrow size distribution (i.e. must be more uniform in size).
3. Crystals are packed and stored in atmosphere where the humidity is less than critical humidity.
4. Crystals may be coated with powdery inert material to prevent absorption of moisture like NaCl is coated with magnesia (MgO) or tricalcium phosphate

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DEPARTMENT OF CHEMICAL ENGINEERING

HEAT AND MASS TRANSFER FOR BIOLOGICAL SYSTEMS

SCHA1311

UNIT – 4 PRINCIPLES OF HEAT TRANSFER - SCHA1311

UNIT 4 PRINCIPLES OF HEAT TRANSFER

Introduction to various modes of heat transfer, Conduction- Fourier's law of heat conduction, thermal conductivity,conduction through liquids. Convection, individual and overall heat transfer coefficient, LMTD, radiation.

Introduction

Heat transfer is a science, which deals with the flow of heat from a higher temperature to lower temperature. Heat cannot be stored and it is defined as the energy in transit due to the difference in the temperatures of the hot and cold bodies. The study of heat transfer not only explains how the heat energy transports but also predicts about the rate of heat transfer. When a certain amount of water is evaporated or condensed, the amount of heat transferred in either of the processes is same. However, the rate of heat transfer in both the cases may be different.

At this point, it is very important to understand about the basic information that the phases of a substance (solid, liquid, and gas) are associated with its energy content. In the solid phase, the molecules or atoms are very closely packed to give a rigid structure (fig.1.1a). In the liquid phase, sufficient thermal energy is present, which keeps the molecules sufficiently apart and as a result the rigidity looses (fig.1.1b). In the gas phase, the presence of additional energy results in a complete separation and the molecules or atoms are free to move anywhere in the space (fig.1.1c). It must be noticed that whenever a change in phase occurs, a large amount of energy involves in the transition.

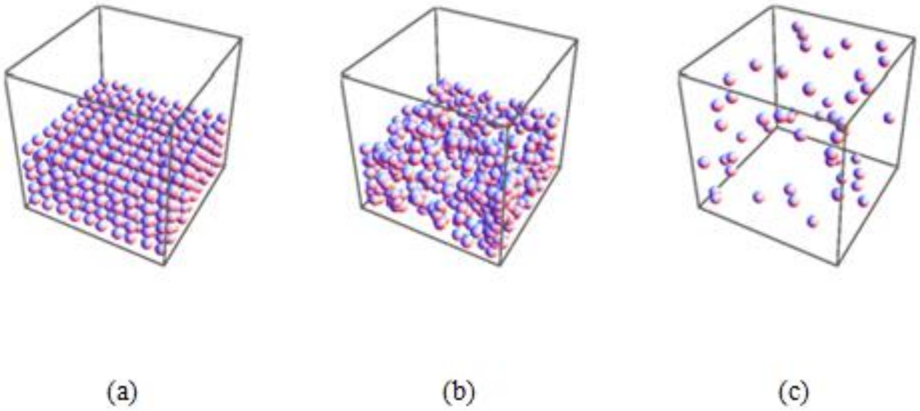


Fig. 1.1: Relative molecular distance of different phases of a substance at a fixed temperature (a) gas/vapour, (b) liquid, and (c) solid

As we are dealing with the heating and cooling of materials in almost our all the processes, the heat transfer is an indispensable part of any of the industries. Therefore, heat transfer is a common subject in many engineering disciplines, especially mechanical and chemical engineering. Study of heat transfer has a vital role in the chemical process industries. Chemical engineers must have a thorough knowledge of heat transfer principles and their applications.

The study of heat transfer is directed to (i) the estimation of rate of flow of energy as heat through the boundary of a system both under steady and transient conditions, and (ii) the determination of temperature field under steady and transient conditions, which also will provide the information about the gradient and time rate of change of temperature at various locations and time. i.e. $T(x, y, z, \tau)$ and dT/dx, dT/dy, dT/dz, $dT/d\tau$ etc. These two are interrelated, one being dependent on the other. However explicit solutions may be generally required for one or the other.

The basic laws governing heat transfer and their application are as below:

1. **First law of thermodynamics postulating the energy conservation principle**: This law provides the relation between the heat flow, energy stored and energy generated in a given system. The relationship for a closed system is: **The net heat flow across the system bondary + heat generated inside the system = change in the internal energy, of the system.** This will also apply for an open system with slight modifications.

The change in internal energy in a given volume is equal to the product of volume density and specific heat ρcV and dT where the group ρcV is called the heat capacity of the system. The basic analysis in heat transfer always has to start with one of these relations.

2. **The second law of thermodynamics** establishing the direction of energy transport as heat. The law postulates that the flow of energy as heat through a system boundary will always be in the direction of lower temperature or along the negative temperature gradient.

3. **Newtons laws of motion** used in the determination of fluid flow parameters.

4. **Law of conservation of mass**, used in the determination of flow parameters.

5. **The rate equations** as applicable to the particular mode of heat transfer.

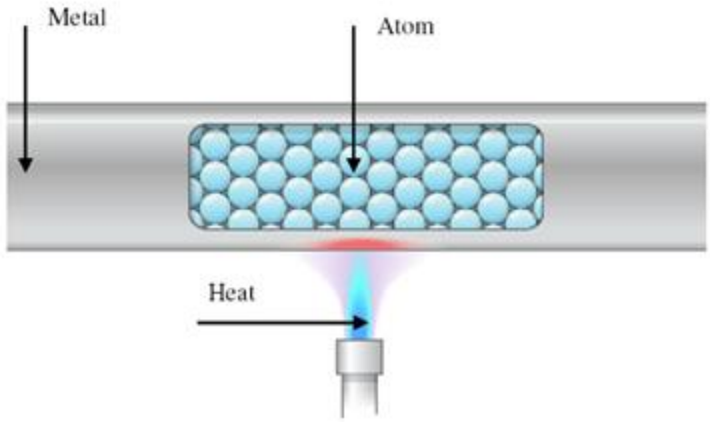
There are three different modes in which heat may pass from a hot body to a cold one. These modes are conduction, convention, and radiation. It should be noted that the heat transfer takes place in combination of two or three modes in any of the real engineering application. In this chapter, we will briefly discuss about the different modes of heat transfer along with the various basic information that will help us as a building block for further study.

Mode of heat transfer

In this section, we will discuss about the three different modes of heat transfer. The discussion will help us to understand about the conduction, convection, and radiation. Moreover, we would be able to understand the basic difference between the three modes of heat transfer.

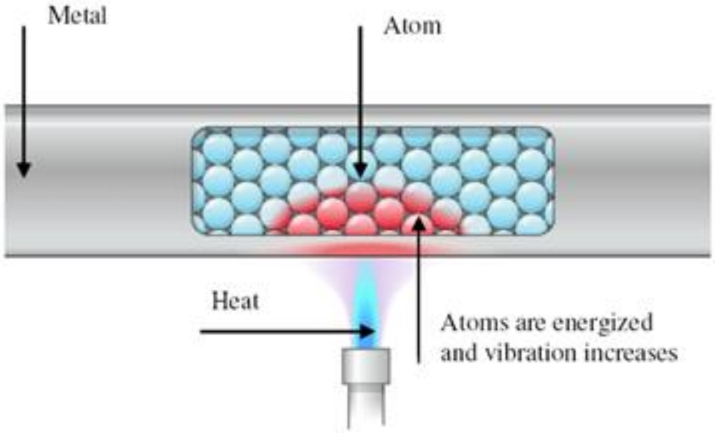
Conduction

Conduction is the transfer of heat in a continuous substance without any observable motion of the matter. Thus, heat conduction is essentially the transmission of energy by molecular motion. Consider a metallic rod being heated at the end and the other end of the rod gets heated automatically. The heat is transported from one end to the other end by the conduction phenomenon. The molecules of the metallic rod get energy from the heating medium and collide with the neighbouring molecules. This process transfers the energy from the more energetic molecules to the low energetic molecules. Thus, heat transfer requires a temperature gradient, and the heat energy transfer by conduction occurs in the direction of decreasing temperature. Figure 1.2 shows an illustration for the conduction, where the densely packed atoms of the rod get energized on heating and vibration effect transfers the heat as described in fig.1.2.



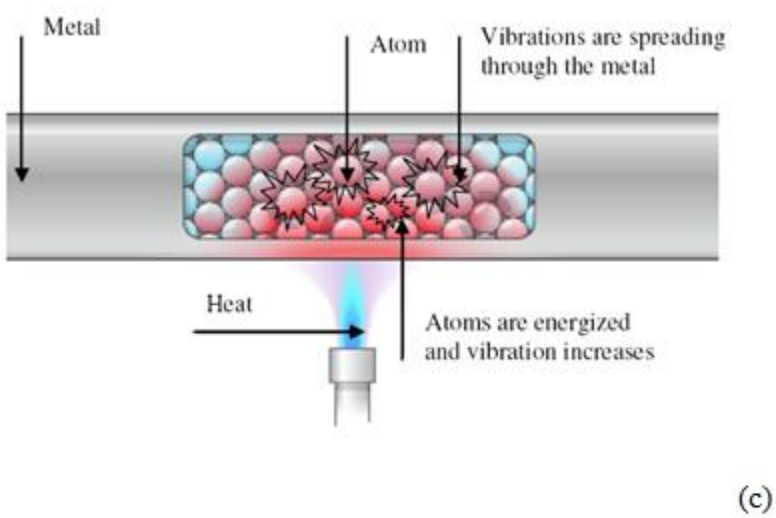
Step 1: The metallic rod is being heated

(a)



Step 2: The energy is transferred to the atoms and vibration of the atoms started

(b)



Step 3: As more energy is transferred to the atoms, the vibration increases as well as transferred to the other atoms

Fig.1.2: Different stages during conduction in a metallic rod

Convection

When a macroscopic particle of a fluid moves from the region of hot to cold region, it carries with it a definite amount of enthalpy. Such a flow of enthalpy is known as convection. Convection may be natural or forced. In natural convection, the movement of the fluid particles is due to the buoyancy forces generated due to density difference of heated and colder region of the fluid as shown in the fig.1.3a. Whereas, in forced convection the movement of fluid particles from the heated region to colder region is assisted by some mechanical means too (eg., stirrer) as shown in fig.1.3b.

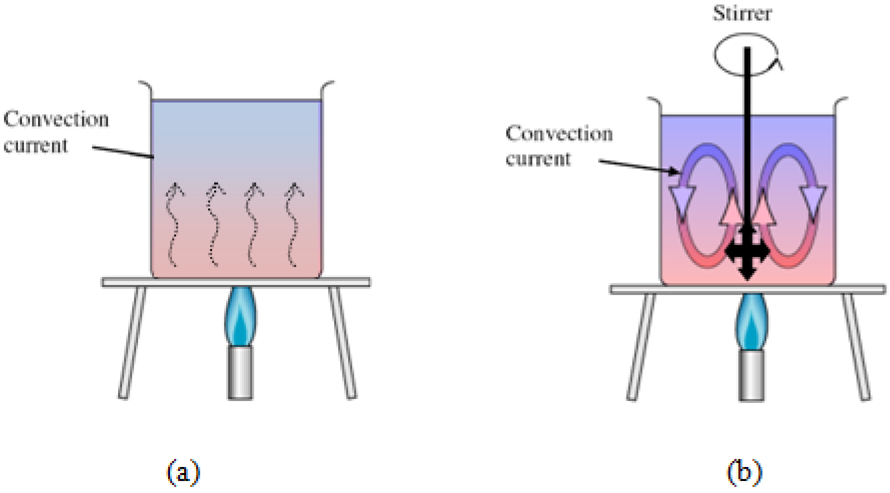


Fig.1.3: Heat transfer through convection (a) natural, and (b) forced

Radiation

We have seen that a medium is required for the heat transfer in case of conduction and convection. However, in case of radiation, electromagnetic waves pass through the empty space. Electromagnetic waves travel at the velocity of light in vacuum. These waves are absorbed, reflected, and/or transmitted by the matter, which comes in the path of the wave. We will limit our discussion (in this NPTEL course) to the thermal radiation. Thermal radiation is the term used to describe the electromagnetic radiation, which is observed to be emitted by the surface of the thermally excited body. The heat of the Sun is the most obvious example of thermal radiation.

There will be a continuous interchange of energy between two radiating bodies, with a net exchange of energy from the hotter to the colder body as shown in the fig.1.4.

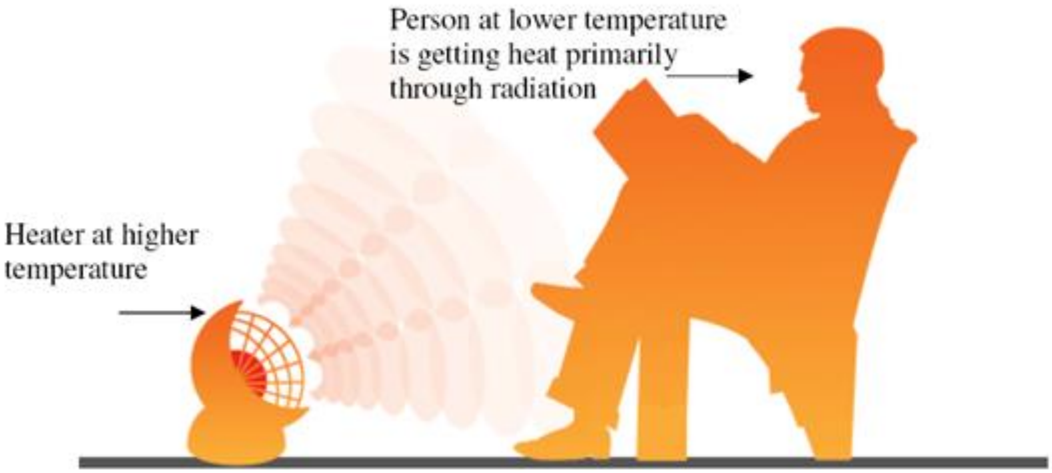


Fig.1.4: Heat transfer through radiation

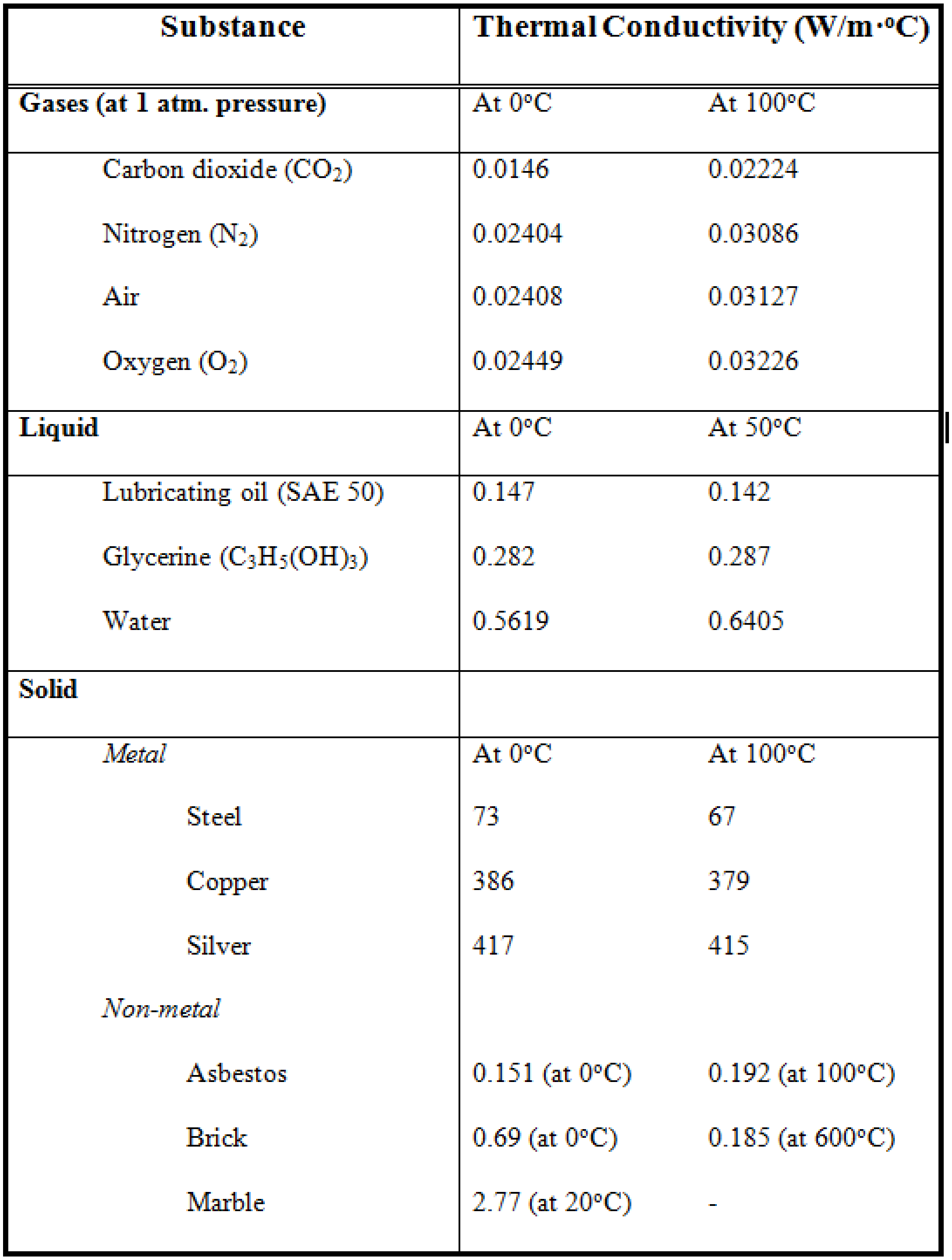
Material properties of importance in heat transfer

Before understanding heat transfer laws, we have to understand various properties of the material. This section is devoted to a brief discussion of some of the important properties of the material.

Thermal conductivity

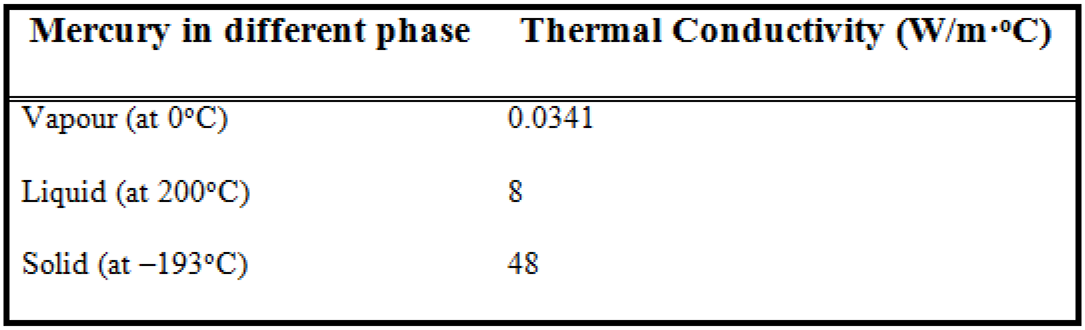
As discussed earlier, the heat conduction is the transmission of energy by molecular action. Thermal conductivity is the property of a particular substance and shows the ease by which the process takes place. Higher the thermal conductivity more easily will be the heat conduction through the substance. It can be realized that the thermal conductivity of a substance would be dependent on the chemical composition, phase (gas, liquid, or solid), crystalline structure (if solid), temperature, pressure, and its homogeneity.
The thermal conductivity of various substances is shown in table-1.1 and table 1.2.

Table-1: Thermal conductivities of various substances at 0oC



Substance	Thermal Conductivity (W/m·°C)	
Gases (at 1 atm. pressure)	At 0°C	At 100°C
Carbon dioxide (CO_2)	0.0146	0.02224
Nitrogen (N_2)	0.02404	0.03086
Air	0.02408	0.03127
Oxygen (O_2)	0.02449	0.03226
Liquid	At 0°C	At 50°C
Lubricating oil (SAE 50)	0.147	0.142
Glycerine ($C_3H_5(OH)_3$)	0.282	0.287
Water	0.5619	0.6405
Solid		
Metal	At 0°C	At 100°C
Steel	73	67
Copper	386	379
Silver	417	415
Non-metal		
Asbestos	0.151 (at 0°C)	0.192 (at 100°C)
Brick	0.69 (at 0°C)	0.185 (at 600°C)
Marble	2.77 (at 20°C)	-

Table-2: Thermal conductivity of mercury at three different phases



Mercury in different phase	Thermal Conductivity (W/m·°C)
Vapour (at 0°C)	0.0341
Liquid (at 200°C)	8
Solid (at −193°C)	48

The general results of the careful analysis of the table-1.1 and 1.2 are as follows,

- Thermal conductivity depends on the chemical composition of the substance.
- Thermal conductivity of the liquids is more than the gasses and the metals have the highest.
- Thermal conductivity of the gases and liquids increases with the increase in temperature.
- Thermal conductivity of the metal decreases with the increase in temperature.
- Thermal conductivity is affected by the phase change.

These differences can be explained partially by the fact that while in gaseous state, the molecules of a substance are spaced relatively far away and their motion is random. This means that energy transfer by molecular impact is much slower than in the case of a liquid, in which the motion is still random but in liquids the molecules are more closely packed. The same is true concerning the difference between the thermal conductivity of the liquid and solid phases. However, other factors are also important when the solid state is formed.

Solid having a crystalline structure has high thermal conductivity than a substance in an amorphous solid state. Metal, crystalline in structure, have greater thermal conductivity than non-metal (refer table-1.1). The irregular arrangement of the molecules in amorphous solids inhibits the effectiveness of the transfer of the energy by molecular impact. Therefore, the thermal conductivity of the non-metals is of the order of liquids. Moreover, in solids, there is an additional transfer of heat energy resulting from vibratory motion of the crystal lattice as a whole, in the direction of decreasing temperature.

Many factors are known to influence the thermal conductivity of metals, such as chemical composition, atomic structure, phase changes, grain size, temperature, and pressure. Out of the above factors, the temperature, pressure, and chemical composition are the most important. However, if we are interested in a particular material then only the temperature effects has to be accounted for.

As per the previous discussion and the table it is now clear that the thermal conductivity of the metal is directly proportional to the absolute temperature and mean free path of the molecules. The mean free path decreases with the increase in temperature so that the thermal conductivity decreases with the temperature. It should be noted that it is true for the pure metal, and the presence of impurity in the metal may reverse the trend. It is usually possible to represent the thermal conductivity of a metal by a linear relation $k = k_o(1 + bT)$, where k_o is the thermal conductivity of the metal at 0°C, $T$is the absolute temperature, and b is a constant.

In general the thermal conductivity of the liquids is insensitive to the pressure if the pressure is not very close to the critical temperature. Therefore, in liquids (as in solids) the temperature effects on the thermal conductivity are generally considered. Liquids, in general, exhibit a decreasing thermal conductivity with temperature. However, water is a notable exception. Water has the highest thermal conductivity among the non-metallic liquids, with a maximum value occurring at 450°C.

The thermal conductivity of a gas is relatively independent of pressure if the pressure is near 1 atm. Vapours near the saturation point show strong pressure dependence. Steam and air are of great engineering importance. Steam shows irregular behaving rather showing a rather strong pressure dependence for the thermal conductivity as well as temperature dependence.

The above discussions concerning thermal conductivity were restricted to materials composed of homogeneous or pure substances. Many of the engineering materials encountered in practice are not of this nature like building material, and insulating material. Some material may exhibit non-isotropic conductivities. The non-isotropic material shows different conductivity in different direction in the material. This directional preference is primarily the result of the fibrous nature of the material like wood, asbestos etc.

Specific heat capacity

Now we know that the thermal conductivity facilitates the heat to propagate through the material due to the temperature gradient. Similarly, specific heat capacity or specific heat is the capacity of heat stored by a material due to variation in temperature. Thus the specific heat capacity (unit: kJ/kg·°C) is defined as the amount of thermal energy required to raise the temperature of a unit amount of material by 1°C. Since heat is path dependent, so is specific heat. In general, the heat transfer processes used in the chemical process plant are at constant pressure; hence the specific heat capacity (c_o) is generally used.

Conduction: One Dimensional

The fundamentals of heat conduction were established over one and a half century and its contribution goes to a French mathematician and physicist, Jean Baptiste Joseph Fourier. You may be aware that any flow whether it is electricity flow, fluid flow, or heat flow needs a driving force. The flow is proportional to the driving force and for various kinds of flows the driving force is shown in the table 3.

Table 3. Various flows and their driving forces

Flow	**Driving force**
Electricity flow	Electric potential gradient
Fluid flow	Pressure gradient
Heat flow	Temperature gradient

Thus the heat flow per unit area per unit time (heat flux, $\dot{q}'$) can be represented by the following relation,

$$\dot{q}' \propto \frac{dT}{dx} \tag{2.1}$$

$$\dot{q}' = k\frac{dT}{dx} \tag{2.2}$$

where, proportionality constant *k* is the thermal conductivity of the material, *T* is the temperature and *x* is the distance in the direction of heat flow. This is known as Fourier's law of conduction.

The term steady-state conduction is defined as the condition which prevails in a heat conducting body when temperatures at fixed points do not change with time. The term one-dimensional is applied to a heat conduction problem when only one coordinate is required to describe the distribution of temperature within the body. Such a situation hardly exists in real engineering problems. However, by considering one-dimensional assumption the real problem is solved fairly upto the accuracy of practical engineering interest.

Steady-state conduction through constant area

A simple case of steady-state, one-dimensional heat conduction can be considered through a flat wall as shown in the fig.2.1.

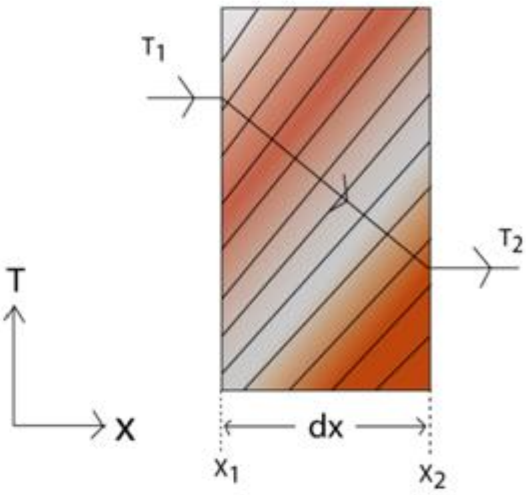


Fig.2.1: Steady-state conduction through a slab (constant area)

The flat wall of thickness d_x is separated by two regions, the one region is at high temperature (T_1) and the other one is at temperature T_2. The wall is very large in comparison of the thickness so that the heat losses from the edges are negligible. Consider there is no generation or accumulation of the heat in the wall and the external surfaces of the wall are at isothermal temperatures T_1 and T_2. The area of the surface through which the heat transfer takes place is A. Then the eq.2.2 can be written as,

$$\dot{q}' = -k\frac{dT}{dx} \tag{2.3}$$

$$\frac{\dot{q}}{A} = -k\frac{dT}{dx} \tag{2.4}$$

$$\frac{\dot{q}}{A} = -k\frac{T_2 - T_1}{x_2 - x_1} \tag{2.5}$$

The negative sign shows that the heat flux is from the higher temperature surface to the lower temperature surface and is the rate of heat transfer through the wall.

Now if we consider a plane wall made up of three different layers of materials having different thermal conductivities and thicknesses of the layers, the analysis of the conduction can be done as follows.

Consider the area (A) of the heat conduction (fig.2.2) is constant and at steady state the rate of heat transfer from layer-1 will be equal to the rate of heat transfer from layer-2. Similarly, the rate of heat transfer through layer-2 will be equal to the rate of heat transfer through layer-3. If we know the surface temperatures of the wall are maintained at T_1 and T_2 as shown in the fig.2.2, the temperature of the interface of layer1 and layer 2 is assumed to be at T' and the interface of layer-2 and layer-3 asT''.

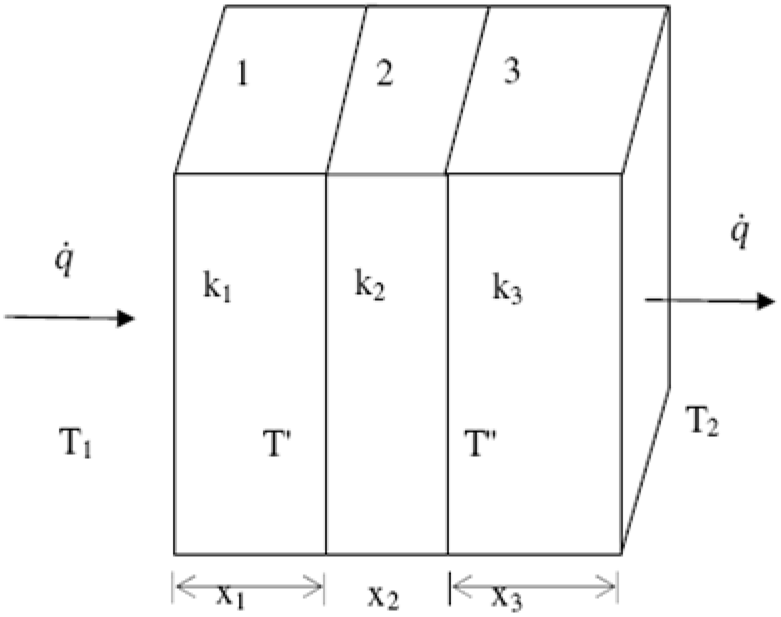


Fig.2.2: Heat conduction through three different layers

The rate of heat transfer through layer-1 to layer-2 will be,

$$\dot{q} = \frac{k_1 A\,(T_1 - T')}{x_1} \quad or \quad (T_1 - T') = \frac{\dot{q}}{1/(x_1/k_1 A)} \qquad (2.6)$$

and,
The rate of heat transfer through layer 2 to layer 3 will be,

$$\dot{q} = \frac{k_2 A\,(T' - T")}{x_2} \quad or \quad (T' - T") = \frac{\dot{q}}{1/(x_2/k_2 A)} \qquad (2.7)$$

and,
The rate of heat transfer through layer 3 to the other side of the wall,

$$\dot{q} = \frac{k_3 A\,(T" - T_2)}{x_3} \quad or \quad (T" - T_2) = \frac{\dot{q}}{1/(x_3/k_3 A)} \qquad (2.8)$$

On adding the above three equations,

$$\dot{q} = \frac{T_1 - T_2}{\frac{x_1}{k_1 A} + \frac{x_2}{k_2 A} + \frac{x_3}{k_3 A}} \qquad (2.9)$$

$$\dot{q} = \frac{T_1 - T_2}{R_1 + R_2 + R_3} \qquad (2.10)$$

Where, R represents the thermal resistance of the layers. The above relation can be written analogous to the electrical circuit as,

$$Rate\ of\ heat\ flow = \frac{Temperaure\ difference}{Resistance} \qquad (2.11)$$

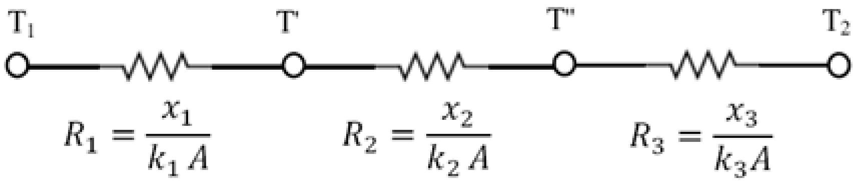


Fig 2.3: Equivalent electrical circuit of the fig.2.2

The wall is composed of 3-different layers in series and thus the total thermal resistance was represented by R (= $R_1 + R_2 + R_3$). The discussed concept can be understood by the illustrations shown below.

The unit of the various parameters used above is summarized as follows,

Parameter	Symbol used	Unit
Heat flux	$\dot{q}'$	W/m^2 or $J/(s \cdot m^2)$
Heat flow rate	$\dot{q}$	W or J/s
Thermal conductivity	k	W/(m·°C)
Thermal resistance	R	°C/W

Lecture 4

Illustration 2.1

The two sides of a wall (2 mm thick, with a cross-sectional area of 0.2 m2) are maintained at 30°C and 90°C. The thermal conductivity of the wall material is 1.28 W/(m·°C). Find out the rate of heat transfer through the wall?

Solution 2.1

Assumptions

1. Steady-state one-dimensional conduction
2. Thermal conductivity is constant for the temperature range of interest
3. The heat loss through the edge side surface is insignificant
4. The layers are in perfect thermal contact

Given,

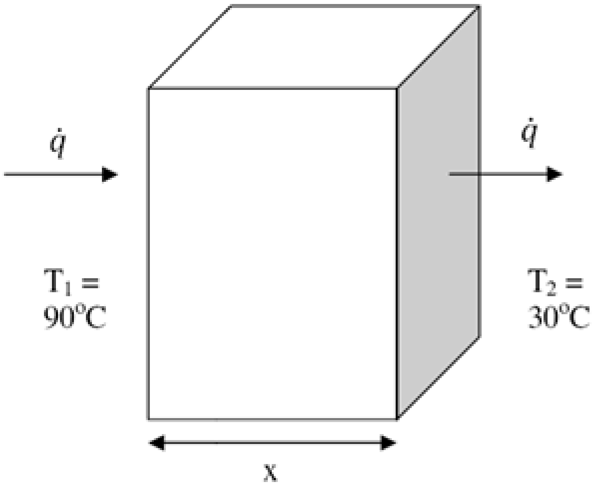


Fig. 2.4: Illustration 2.1

$k = 1.28$ W/(m·°C) $A = 0.2\ m^2$ $x = 2$ mm$=0.002$ m

The rate of heat transfer can be written as,

$$\dot{q} = \frac{\Delta T}{x/_{kA}}$$

$$\dot{q} = \frac{90 - 30}{0.002/_{1.28 \times 0.2}} = 7680\,W$$

Illustration 2.2

One side of a 1 cm thick stainless steel wall (k_1 = 19 W/m·°C) is maintained at 180°C and the other side is insulated with a layer of 4 cm fibreglass (k_2 = 0.04 W/m·°C). The outside of the fibreglass is maintained at 60°C and the heat loss through the wall is 300 W. Determine the area of the wall?

Solution 2.2

Assumptions:

1. Steady-state one-dimensional conduction.
2. Thermal conductivity is constant for the temperature range of interest.
3. The heat loss through the edge side surface is insignificant.
4. The layers are in perfect thermal contact.

Given, $k_1 = 19$ W/m·°C $k_2 = 0.04$ W/m·°C $\dot{q} = 300$ W

$x_1 = 1$ cm $= 0.01$ m $x_2 = 4$ cm $= 0.04$ m

The resistance of the above composite,

$$R = \frac{x_1}{k_1\,A} + \frac{x_2}{k_2\,A}$$

On putting all the known values,

$$R = \frac{0.01}{19\;A} + \frac{0.04}{0.04\;A}$$

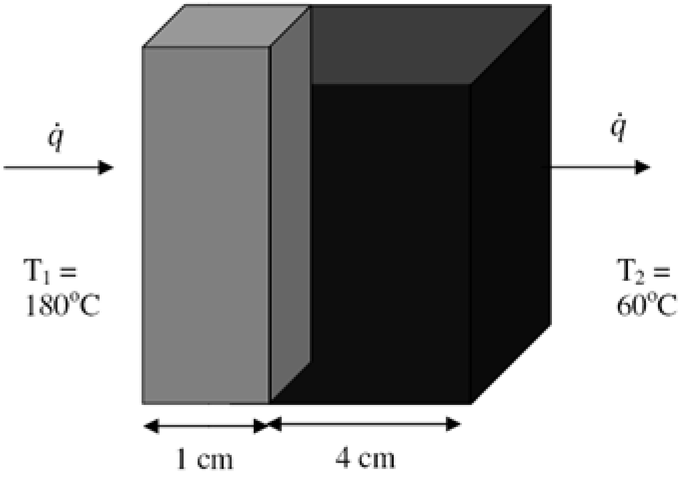


Fig. 2.5: Illustration 2.2

Thus,

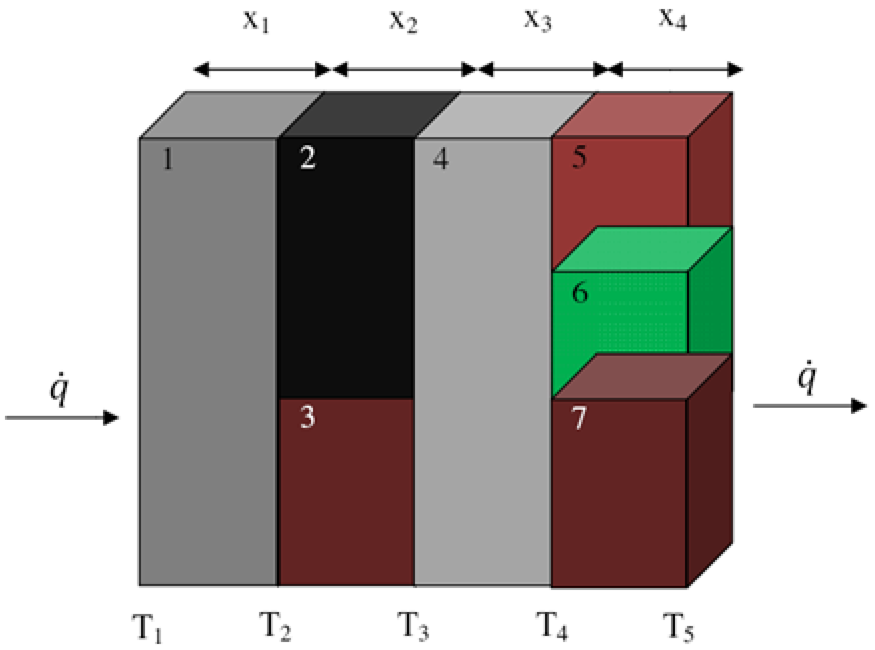
$$\dot{q} = \frac{T_1 - T_2}{R}$$

$$300 = \frac{180 - 60}{\frac{0.01}{19\,A} + \frac{0.04}{0.04\,A}}$$

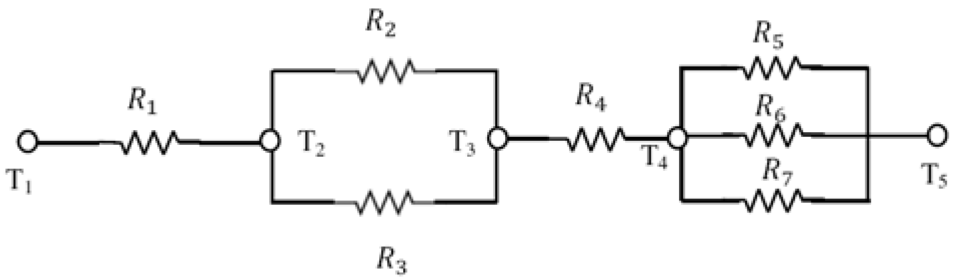
On solving, $A = 2.5\ m^2$

The previous discussion showed the resistances of different layers. Now to understand the concept of equivalent resistance, we will consider the geometry of a composite as shown in fig.2.6a.

The wall is composed of seven different layers indicated by 1 to 7. The interface temperatures of the composite are T_1 to T_5 as shown in the fig.2.6a. The equivalent electrical circuit of the above composite is shown in the fig 2.6b below,



(a)



(b)

Fig.2.6. (a) Composite wall, and (b) equivalent electrical circuit

The equivalent resistance of the wall will be,

$$R = R_1 + \frac{1}{{}^{1}/_{R_2} + {}^{1}/_{R_3}} + R_4 + \frac{1}{{}^{1}/_{R_5} + {}^{1}/_{R_6} + {}^{1}/_{R_7}} \qquad (2.12)$$

where,

$$R_i = \frac{x_i}{k_i\, A_i}$$

Therefore, at steady state the rate of heat transfer through the composite can be represented by,

$$\dot{q} = \frac{T_1 - T_5}{R} \qquad (2.13)$$

where, R is the equivalent resistance.

Illustration **2.3**

Consider a composite wall containing 5-different materials as shown in the fig. 2.7. Calculate the rate of heat flow through the composite from the following data?

$x_1 = 0.1\ m$
$x_2 = 0.2\ m$
$x_3 = 0.15\ m$
$k_1 = 15\ W/m{\cdot}^oC$
$k_2 = 25\ W/m{\cdot}^oC$
$k_3 = 30\ W/m{\cdot}^oC$
$k_4 = 20\ W/m{\cdot}^oC$
$k_5 = 35\ W/m{\cdot}^oC$
$h_2 = 1\ m$
$h_3 = 3\ m$
$h_4 = 2.5\ m$
$h_5 = 1.5\ m$
$T_A = 120^oC$
$T_B = 50^oC$

Solution 2.3

Assumptions:

1. Steady-state one-dimensional conduction.
2. Thermal conductivity is constant for the temperature range of interest.
3. The heat loss through the edge side surface is insignificant.
4. The layers are in perfect thermal contact.
5. Area in the direction of heat flow is 1 m^2.

The height of the first layer is 4 m ($h_1 = h_2 + h_3$).
The equivalent circuit diagram of the above composite is,

$$R = R_1 + \frac{1}{{}^{1}/_{R_2} + {}^{1}/_{R_3}} + \frac{1}{{}^{1}/_{R_4} + {}^{1}/_{R_5}}$$

On calculating equivalent resistance with the given data (Note: thickness of layer 2 = thickness of layer 3 and thickness of layer 4 = thickness of layer 5, in the heat flow direction),

$$R = \frac{0.1}{(15)(1)} + \frac{1}{\frac{1}{\left(\frac{0.2}{25\left(\frac{1}{4}\right)}\right)} + \frac{1}{\left(\frac{0.2}{30\left(\frac{3}{4}\right)}\right)}} + \frac{1}{\frac{1}{\left(\frac{0.15}{20\left(\frac{2.5}{4}\right)}\right)} + \frac{1}{\left(\frac{0.15}{35\left(\frac{1.5}{4}\right)}\right)}}$$

$$R = 0.0195\,\frac{°\mathrm{C}}{W}$$

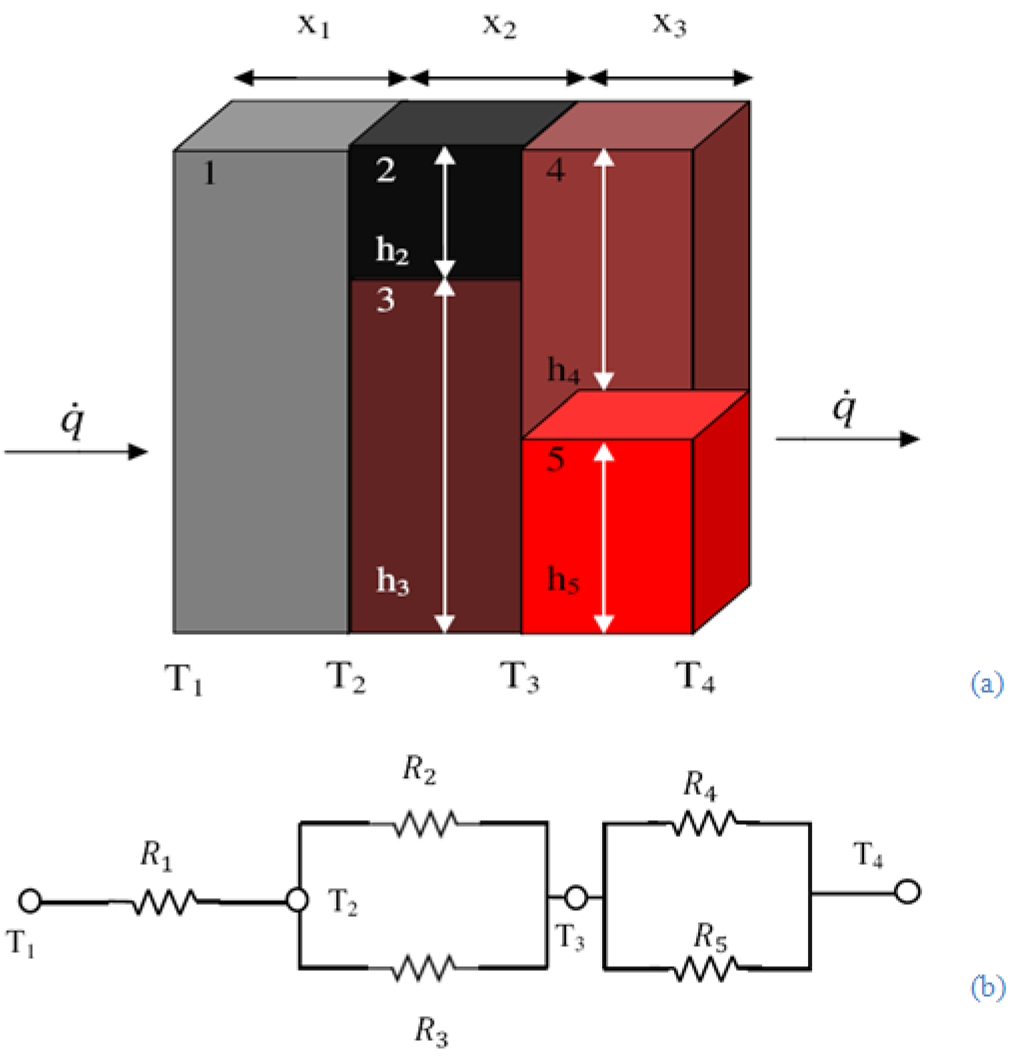


Fig. 2.7: Composite of illustration 2.3; (a) composite, (b) corresponding electrical circuit

Thus the heat flow rate through the composite,

$$\dot{q} = \frac{T_A - T_B}{R}$$

$$\dot{q} = \frac{120 - 50}{0.0195} = 3.59\ kW$$

2.2 Thermal contact resistance

In the previous discussion, it was assumed that the different layers of the composite have perfect contact between any two layers. Therefore, the temperatures of the layers were taken same at the plane of contact. However, in reality it rarely happens, and the contacting surfaces are not in perfect contact or touch as shown in the fig.2.8(a). It is because as we know that due to the roughness of the surface, the solid surfaces are not perfectly smooth. Thus when the solid surfaces are contacted the discrete points of the surfaces are in contact and the voids are generally filled with the air. Therefore, the heat transfer across the composite is due to the parallel effect of conduction at solid contact points and by convection or probably by radiation (for high temperature) through the entrapped air. Thus an apparent temperature drop may be assumed to occur between the two solid surfaces as shown in the fig.2.8b. If T_I and T_{II} are the theoretical temperature of the plane interface, then the thermal contact resistance may be defined as,

$$R_c = \frac{T_I - T_{II}}{\dot{q}} \qquad (2.14)$$

where $R_c$represents the thermal contact resistance.

The utility of the thermal contact resistance (R_c) is dependent upon the availability of the reliable data. The value of R_c depends upon the solids involved, the roughness factor, contact pressure, material occupying the void spaces, and temperature. The surface roughness of a properly smooth metallic surface is in the order of micrometer. The values of R_c generally obtained by the experiments. However, there are certain theories which predict the effect of the various parameters on the R_c.

It can be seen in the fig.2.8, that the two main contributors to the heat transfer are (i) the conduction through entrapped gases in the void spaces and, (ii) the solid-solid conduction at the contact points. It may be noted that due to main contribution to the resistance will be through first factor because of low thermal conductivity of the gas.

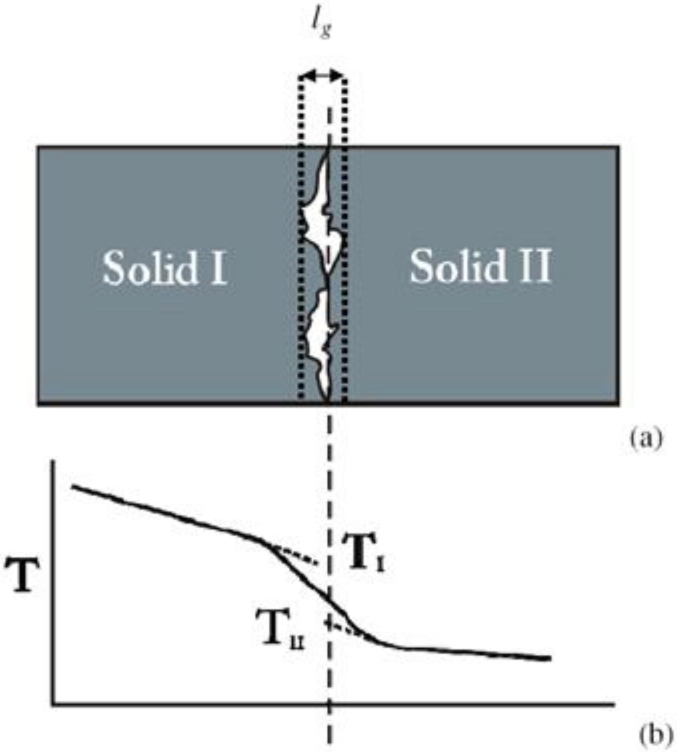


Fig.2.8 (a) Contacting surfaces of two solids are not in perfect contact, (b) temperature drop due to imperfect contact

If we denote the void area in the joint by A_v and contact area at the joint by A_c, then we may write heat flow across the joint as,

$$\dot{q} = \frac{T_I - T_{II}}{\frac{l_g}{2k_I A_C}+\frac{l_g}{2k_{II} A_C}} + k_f A_v \frac{T_I - T_{II}}{l_g}$$

$$\frac{T_I - T_{II}}{R_C} = \frac{T_I - T_{II}}{\frac{l_g}{2k_I A_C}+\frac{l_g}{2k_{II} A_C}} + k_f A_v \frac{T_I - T_{II}}{l_g}$$

where, thickness of the void space and thermal conductivity of the fluid (or gas) is represented by $l_g$and k_f, respectively. It was assumed that $l_g/2$ is the thickness of solid-I and solid-II for evenly rough surfaces.

Lecture 62.3 Steady-state heat conduction through a variable area

It was observed in the previous discussion that for the given plane wall the area for heat transfer was constant along the heat flow direction. The plane solid wall was one of the geometries but if we take some other geometry (tapered plane, cylindrical body, spherical body etc.) in which the area changes in the direction of heat flow. Now we will consider geometrical configuration which will be mathematically simple and also of great engineering importance like hollow cylinder and hollow sphere. In these cases the heat transfer area varies in the radial direction of heat conduction. We will take up both the cases one by one in the following sections.

2.3.1 Cylinder

Consider a hollow cylinder as shown in the fig.2.9a. The inner and outer radius is represented by $r_i$and r_o , whereas T_i and T_o ($T_i > T_o$) represent the uniform temperature of the inner and outer wall, respectively.

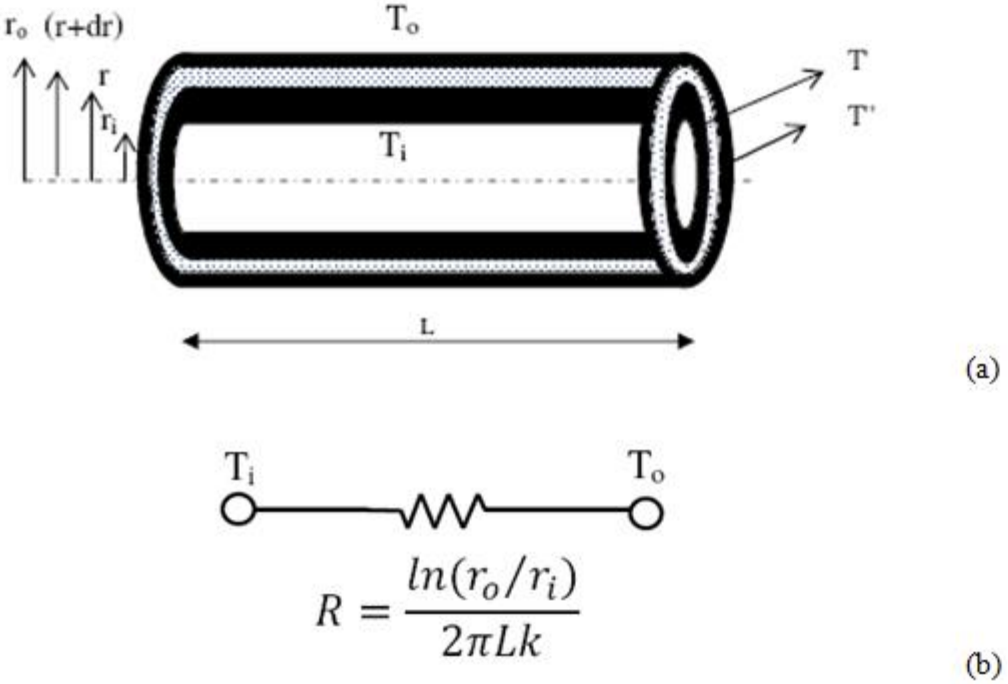


Fig. 2.9. (a) Hollow cylinder, (b) equivalent electrical circuit

Consider a very thin hollow cylinder of thicknessd_r in the main geometry (fig.2.9a) at a radial distance$r$. If d_r is small enough with respect to r, then the area of the inner and outer surface of the thin cylinder may be considered to be of same area. In other words, for very small d_r with respect to r, the lines of heat flow may be considered parallel through the differential element in radial outward direction.

We may ignore the heat flow through the ends if the cylinder is sufficiently large. We may thus eliminate any dependence of the temperature on the axial coordinate and for one dimensional steady state heat conduction, the rate of heat transfer for the thin cylinder,

$$\dot{q} = -k\frac{dT}{dr}\,2\pi rL$$

Where dT is the temperature difference between the inner and outer surface of the thin cylinder considered above and k is the thermal conductivity of the cylinder. On rearranging,

$$\frac{dr}{r} = -\frac{2\pi Lk}{\dot{q}} dT$$

To get the heat flow through the thick wall cylinder, the above equation can be integrated between the limits,

$$\int_{r_i}^{r_o} \frac{dr}{r} = -\frac{2\pi Lk}{\dot{q}} \int_{T_i}^{T_o} dT$$

On solving,

$$\dot{q} = k(2\pi L)\frac{(T_i - T_o)}{ln(r_o/r_i)} \tag{2.15}$$

$$\dot{q} = kA_{LM}\frac{(T_i - T_o)}{r_o - r_i} \tag{2.16}$$

Where $A_{LM} = \frac{2\pi L\,(r_o - r_i)}{ln(r_o/r_i)}$, and the careful analysis of the above equation shows that the expression is same as for heat flow through the plane wall of thickness (r_o–r_i) except the expression for the area. The A_{LM} is known as log mean area of the cylinder, whose length is L and radius is r_{LM} (= $\frac{(r_o - r_i)}{ln(r_o/r_i)}$). The fig.2.9b shows the equivalent electrical circuit of the fig.2.9b.

Now we have learnt that how to represent the analogous electrical circuit for the cylindrical case. It will provide the building block for the composite cylinders similar to the plane composite we have learnt earlier. The following fig.2.10a shows a composite cylinder with 4-layers of solid material of different inner and outer diameter as well as thermal conductivity. The equivalent electrical circuit is shown below in fig.2.10b.

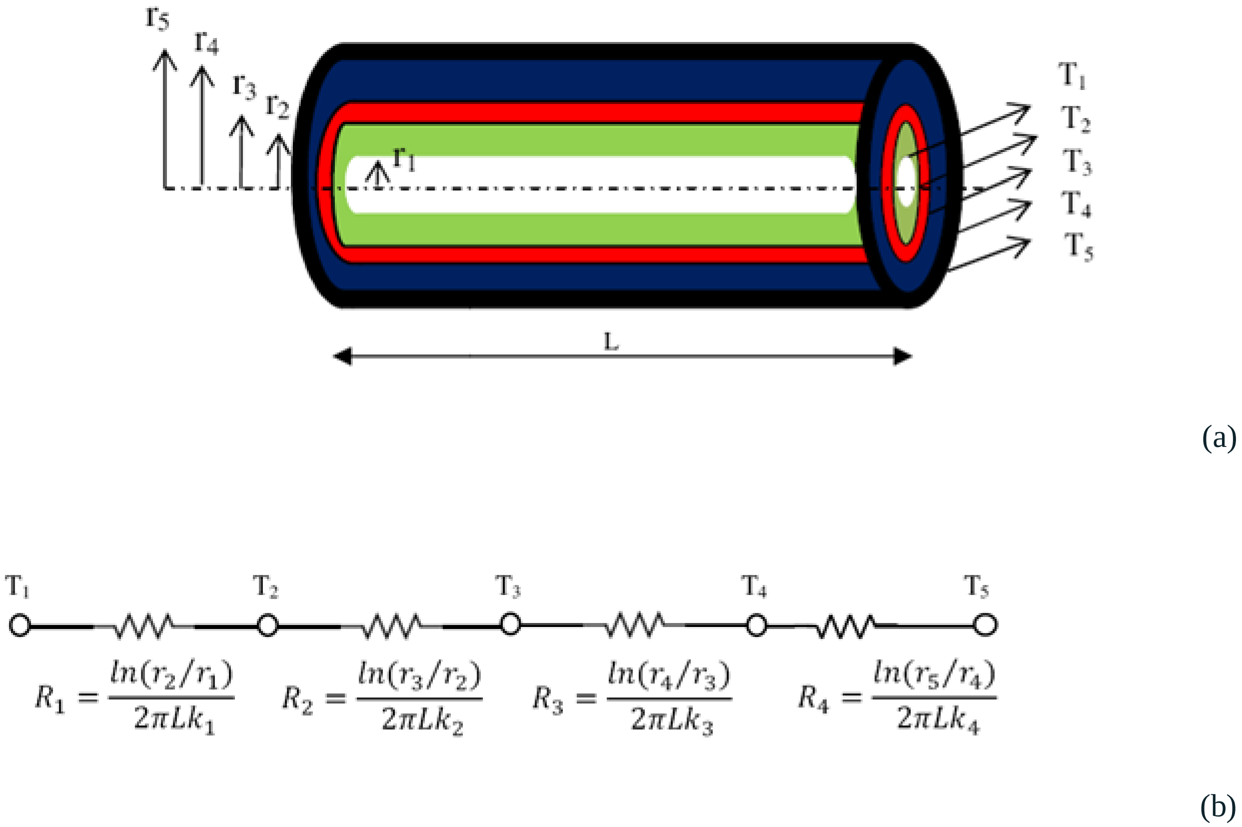


Fig.2.10.(a) Four layer composite hollow cylinder, (a) equivalent electrical circuit

The total heat transfer at steady-state will be,

$$\dot{q} = \frac{T_1 - T_5}{R_1 + R_2 + R_3 + R_4} \qquad (2.17)$$

where R_1 , R_2 , R_3 , and R_4 are represented in the fig.2.10b.

2.3.2 Sphere

The rate of heat transfer through a hollow sphere can be determined in a similar manner as for cylinder. The students are advised to derive the following expression shown below.

The final expression for the rate of heat flow is,

$$\dot{q} = 4\pi k \frac{(T_i - T_o)}{1/r_o - 1/r_i} \qquad (2.18)$$

convection.

The rate of heat transfer in a solid body or medium can be calculated by Fourier's law. Moreover, the Fourier law is applicable to the stagnant fluid also. However, there are hardly a few physical situations in which the heat transfer in the fluid occurs and the fluid remains stagnant. The heat transfer in a fluid causes convection (transport of fluid elements) and thus the heat transfer in a fluid mainly occurs by convection.

3.1 Principle of heat flow in fluids and concept of heat transfer coefficient

It is learnt by day-to-day experience that a hot plate of metal will cool faster when it is placed in front of a fan than exposed to air, which is stagnant. In the process, the heat is convected away, and we call the process convective heat transfer. The term convective refers to transport of heat (or mass) in a fluid medium due to the motion of the fluid. Convective heat transfer, thus, associated with the motion of the fluid. The term convection provides an intuitive concept of the heat transfer process. However, this intuitive concept must be elaborated to enable one to arrive at anything like an adequate analytical treatment of the problem.

It is well known that the velocity at which the air blows over the hot plate influences the heat transfer rate. A lot of questions come into the way to understand the process thoroughly. Like, does the air velocity influence the cooling in a linear way, i.e., if the velocity is doubled, will the heat transfer rate double. We should also suspect that the heat-transfer rate might be different if we cool the plate with some other fluid (say water) instead of air, but again how much difference would there be? These questiones may be answered with the help of some basic analysis in the later part of this module.

The physical mechanism of convective heat transfer for the problem is shown in fig.3.1.

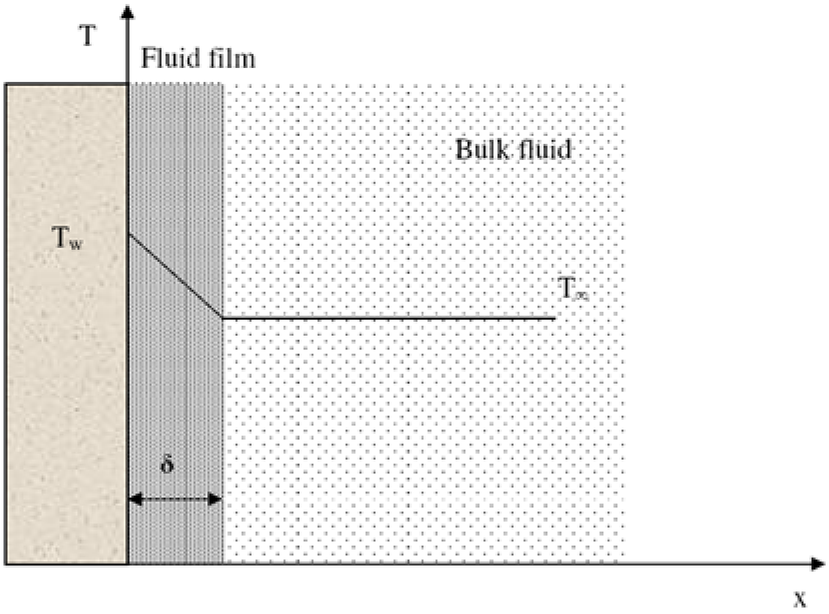


Fig. 3.1: Convective heat transfer from a heated wall to a fluid

Consider a heated wall shown in fig.3.1. The temperature of the wall and bulk fluid is denoted by T_w, and T_∞, respectively. The velocity of the fluid layer at the wall will be zero. Thus the heat will be transferred through the stagnant film of the fluid by conduction only. Thus we can compute the heat transfer using Fourier's law if the thermal conductivity of the fluid and the fluid temperature gradient at the wall is known. Why, then, if the heat flows by conduction in this layer, do we speak of convective heat transfer and need to consider the velocity of the fluid? The answer is that the temperature gradient is dependent on the rate at which the fluid carries the heat away; a high velocity produces a large temperature gradient, and so on. However, it must be remembered that the physical mechanism of heat transfer at the wall is a conduction process.

It is apparent from the above discussion that the prediction of the rates at which heat is convected away from the solid surface by an ambient fluid involves thorough understanding of the principles of heat conduction, fluid dynamics, and boundary layer theory. All the complexities involved in such an analytical approach may be lumped together in terms of a single parameter by introduction of Newton's law of cooling,

$$\frac{\dot{q}}{A} = h\,(T_w - T_\infty) \tag{3.1}$$

where, *h* is known as the heat transfer coefficient or film coefficient. It is a complex function of the fluid composition and properties, the geometry of the solid surface, and the hydrodynamics of the fluid motion.

If *k* is the thermal conductivity of the fluid, the rate of heat transfer can be written directly by following the Fourier's law. Therefore, we have,

$$\dot{q} = -k\,A\,\frac{(T_\infty - T_w)}{\delta} = \frac{k}{\delta}\,A\,(T_w - T_\infty) \tag{3.2}$$

where, $\frac{(T_\infty - T_w)}{\delta}$ is the temperature gradient in the thin film where the temperature gradient is linear.

On comparing eq.3.1 and 3.2, we have,

$$h = \frac{k}{\delta} \tag{3.3}$$

It is clear from the above expression that the heat transfer coefficient can be calculated if k and δ are known. Though the k values are easily available but the δ is not easy to determine. Therefore, the above equation looks simple but not really easy for the calculation of real problems due to non-linearity of k and difficulty in determining δ. The heat transfer coefficient is important to visualize the convection heat transfer phenomenon as discussed before. In fact, δ is the thickness of a heat transfer resistance as that really exists in the fluid under the given hydrodynamic conditions. Thus, we have to assume a film of δ thickness on the surface and the heat transfer coefficient is determined by the properties of the fluid film such as density, viscosity, specific heat, thermal conductivity etc. The effects of all these parameters are lumped or clubbed together to define the film thickness. Henceforth, the heat transfer coefficient (h) can be found out with a large number of correlations developed over the time by the researchers. These correlations will be discussed in due course of time as we will proceed through the modules. Table 3.1 shows the typical values of the convective heat transfer coefficient under different situations.

Table-3.1: Typical values of *h* under different situations

Description	**Heat Transfer Coefficient (W/m^2·°C)**
Free convection in air	5-25
Forced convection in air	10-500
Free convection in water	500-1,000
Forced convection in water	1,000-15,000
Boiling water	2,500-25,000
Condensing water	5,000-1,00,000

3.2 Individual and overall heat transfer coefficient

If two fluids are separated by a thermally conductive wall, the heat transfer from one fluid to another fluid is of great importance in chemical engineering process plant. For such a case the rate of heat transfer is done by considering an overall heat transfer coefficient. However, the overall heat transfer coefficient depends upon so many variables that it is necessary to divide it into individual heat transfer coefficients. The reason for this becomes apparent if the above situation can be elaborated as discussed in the following sub-sections.

3.2.1 Heat transfer between fluids separated by a flat solid wall

As shown in fig.3.2, a hot fluid is separated by solid wall from a cold fluid. The thickness of the solid wall is l, the temperature of the bulk of the fluids on hot and cold sides are T_h and T_c, respectively. The average temperature of the bulk fluid is T_1 and T_4, for hot and cold fluid, respectively. The thicknesses of the fictitious thin films on the hot and cold sides of the flat solid are shown by δ_1 and δ_2. It may be assumed that the Reynolds numbers of both the fluids are sufficiently large to ensure turbulent flow and the surfaces of the solid wall are clean.

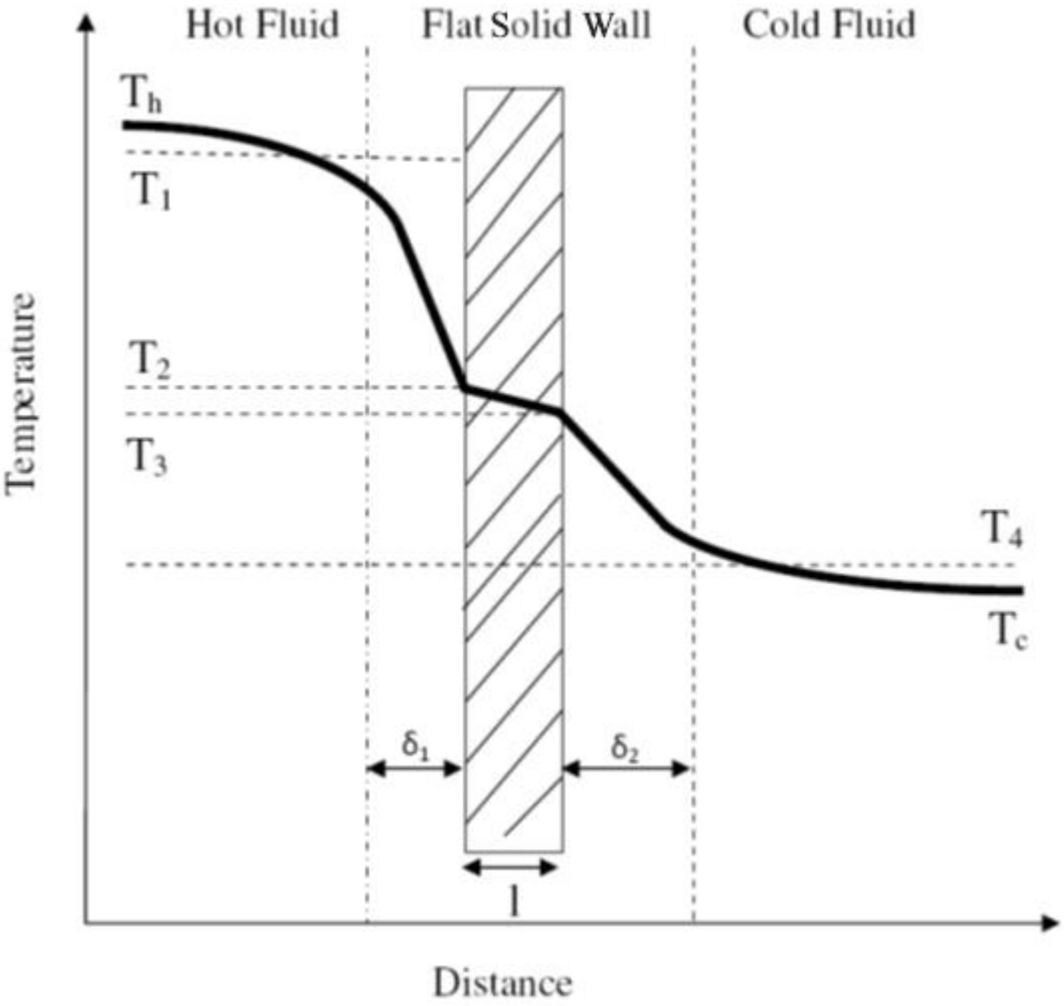


Fig.3.2. Real temperature profile

It can be seen that the temperature gradient is large near the wall (through the viscous sublayer), small in the turbulent core, and changes rapidly in the buffer zone (area near the interface of sublayer and bulk fluid). The reason was discussed earlier that the heat must flow through the viscous sublayer by conduction, thus a steep temperature gradient exists because of the low temperature gradient of most of the fluids.

The average temperatures of the warm bulk fluid and cold bulk fluids are slightly less than the maximum temperature T_h (bulk temperature of hot fluid) and slightly more than the minimum temperature T_c (bulk temperature of cold fluid), respectively. The average temperatures are shown by T_1 and T_4, for the hot and cold fluid streams, respectively.

Figure 3.3 shows the simplified diagram of the above case, where *T2* and *T3* are the temperatures of the fluid wall interface.

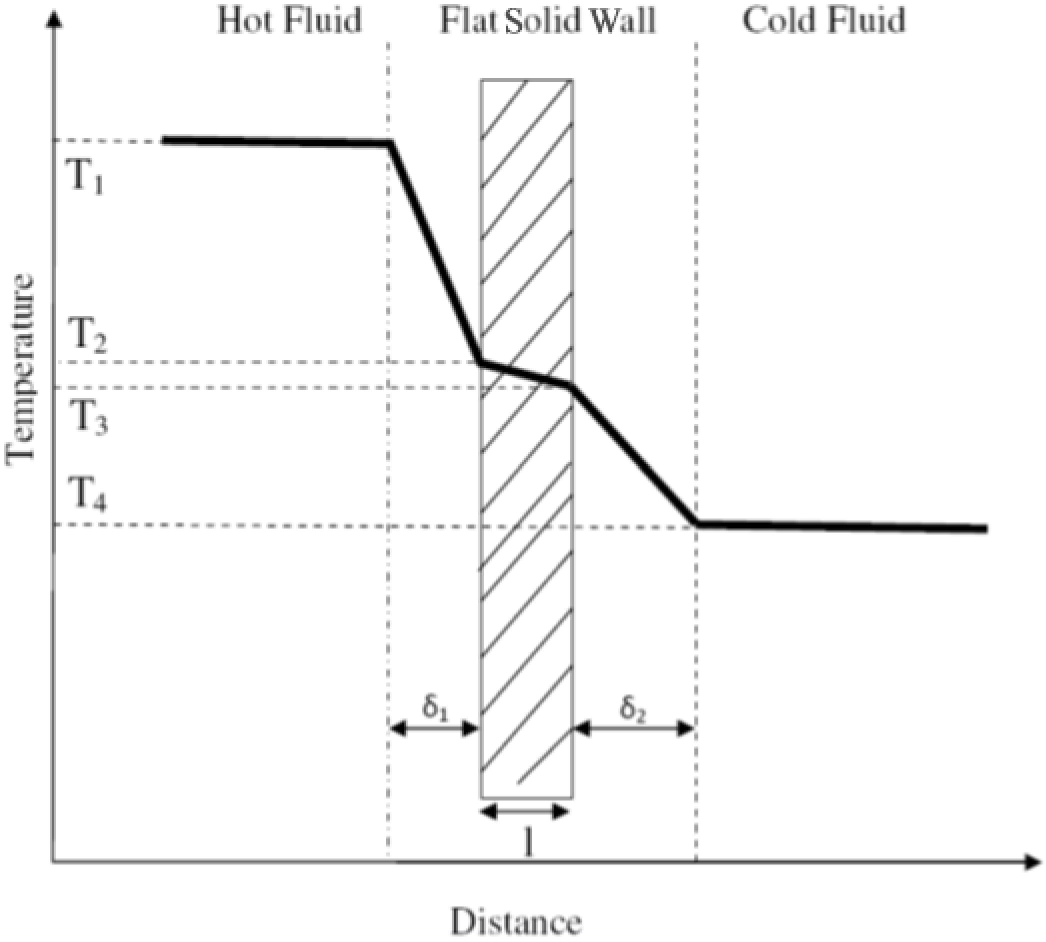


Fig.3.3. Simplified temperature profile for fig.3.2

If the thermal conductivity of the wall is *k*, and the area of the heat transfer is *A*, the electrical analogy of the fig.3.3 can be represented by fig.3.4, where h_1 and h_2 are the individual heat transfer coefficient of the hot and cold side of the fluid.

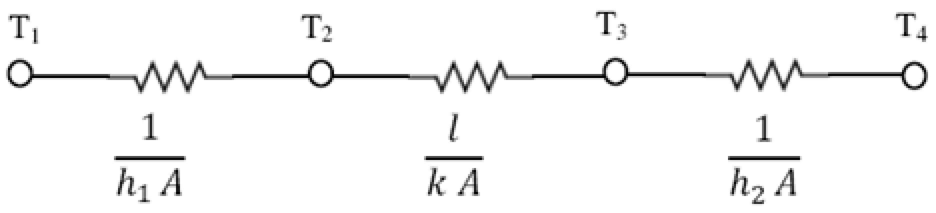


Fig.3.4. Equivalent electrical circuit for fig. 3.3

Considering that the heat transfer is taking place at the steady-state through a constant area and the heat loss from other faces are negligible, then the rate of heat transfer on two sides of the wall will be represented by eq. 3.4-3.6.

Rate of heat transfer from the hot fluid to the wall,

$$\dot{q}_1 = h_1 A\,(T_1 - T_2) \tag{3.4}$$

Rate of heat transfer through the wall,

$$\dot{q}_2 = \frac{k A\,(T_2 - T_3)}{l} \tag{3.5}$$

Rate of heat transfer from the wall to cold fluid,

$$\dot{q}_3 = h_2 A\,(T_3 - T_4) \tag{3.6}$$

At steady state, the rate of heat transfers $(\dot{q}_1, \dot{q}_2, \text{ and } \dot{q}_3)$ are same and can be represented by $\dot{q}$. Therefore,

$$T_1 - T_2 = \frac{\dot{q}}{h_1\, A} \tag{3.7}$$

$$T_2 - T_3 = \frac{\dot{q}}{A\, k/l} \tag{3.8}$$

$$T_2 - T_3 = \frac{\dot{q}}{h_2\, A} \tag{3.9}$$

On adding equations (3.7 to 3.9)

$$T_1 - T_4 = \frac{\dot{q}}{A}\left(\frac{1}{h_1} + \frac{1}{k/l} + \frac{1}{h_3}\right)$$

$$T_1 - T_4 = \frac{\dot{q}}{A}\frac{1}{U} \tag{3.10}$$

where,

$$\frac{1}{U} = \frac{1}{h_1} + \frac{l}{k} + \frac{1}{h_3}$$

Thus,

$$\dot{q} = UA(T_1 - T_4) \tag{3.11}$$

The quantity U is called the overall heat transfer coefficient (can be calculated if the $h_1, h_2, k,$ and l are known). Thus from the system described is established that the overall heat transfer coefficient is the function of individual heat transfer coefficient of the fluids on the two sides of the wall, as well as the thermal conductivity of the flat wall. The overall heat transfer coefficient can be used to introduce the controlling term concept. The controlling resistance is a term which possesses much larger thermal resistance compared to the sum of the other resistances. At this point it may be noted that in general the resistance offered by the solid wall is much lower. Similarly, if a liquid and a gas are separated by a solid wall the resistance offered by the gas film may generally be high.

he steady state temperature distribution in a wall is , where x (in meter) is the position in the wall and T is the temperature (in °C). The thickness of the wall is 0.2 m and the thermal conductivity of the wall is 1.2 (W/m·°C). The wall dissipates the heat to the ambient at 30 °C. Calculate the heat transfer coefficient at the surface of the wall at 0.2 m.

Solution **3.1**

The rate of heat transfer through the wall by conduction will be equal to the rate of heat transfer from the surface to the ambient by convention at steady state,

Rate of heat transfer by conduction at x=0.2 is given by,

$$-kA\frac{dT}{dx} = h\,A\,(T_x - T_a)$$

where T_a is the ambient temperature.

$T = 300 - 3050x^2$

$T_{x=0.2} = 300 - 3050(0.2^2) = 178\ °\mathrm{C}$

$$\frac{dT}{dx} = -6100x$$

$$h = \frac{-kA\frac{dT}{dx}}{A(T_x - T_a)}$$

$$= \frac{(-k)(-6100x)}{T_x - T_a}$$

On putting the values and solving,

$$= \frac{(1.2)(6100)(0.2)}{178 - 30}$$

$h = 9.89\ \mathrm{W/(m^2 \cdot °C)}$

Log mean temperature difference (LMDT):

The log mean temperature difference (LMTD) is used to determine the temperature driving force for heat transfer in flow systems, most notably in heat exchangers. The LMTD is a logarithmic average of the temperature difference between the hot and cold streams at each end of the exchanger. The larger the LMTD, the more heat is transferred. The use of the LMTD arises straightforwardly from the analysis of a heat exchanger with constant flow rate and fluid thermal properties.

We assume that a generic heat exchanger has two ends (which we call "A" and "B") at which the hot and cold streams enter or exit on either side; then, the LMTD is defined by the logarithmic mean as follows:

$$LMTD = \frac{\Delta T_A - \Delta T_B}{\ln\left(\frac{\Delta T_A}{\Delta T_B}\right)}$$

where ΔTA is the temperature difference between the two streams at end A, and ΔTB is the temperature difference beween the two streams at end B.

This equation is valid both for parallel flow, where the streams enter from the same end, and for counter-current flow, where they enter from different ends.

Once calculated, the LMTD is usually applied to calculate the heat transfer in an exchanger according to the simple equation:

$$Q = U \times A \times LMTD$$

Where Q = Heat transfer

⇒ Once calculated, the LMTD is usually applied to calculate the heat transfer in an exchanger according to the simple equation:

$$Q = U \times A \times LMTD$$

Where Q is the exchanged heat duty (in watts), U is the heat transfer coefficient (in watts per kelvin per square meter) and A is the exchange area. Note that estimating the heat transfer coefficient may be quite complicated.

It is to be noted that the following assumption have been considered for developing LMTD,

1. The overall heat transfer coefficient is constant throughout the exchanger
2. In case any fluid undergoes for phase change (e.g., in condenser), the phase change occurs throughout the heat exchanger and the constant fluid temperature prevails throughout the exchanger.
3. The specific heat and mass flow rate and hence the heat capacity rate, of each fluid is constant.
4. No heat is lost in to the surroundings.
5. There is no conduction in the direction of flow neither in the fluids nor in the tube or shell walls.
6. Each of the fluids may be characterized by a single temperature, at any cross section in the heat exchanger that is ideal transverse mixing in each fluid is presumed.

F_T, the LMTD correction factor can be directly obtained from available charts in the literature. These charts were prepared from the results obtained theoretically by solving the temperature distribution in multi-pass heat exchangers.

Figures 8.12 and 8.13 show the two generally used heat exchangers and their corresponding plots for finding F_T. It may be noted that the given figures have the representative plots and any standard book on heat transfer may be consulted for the accurate results.

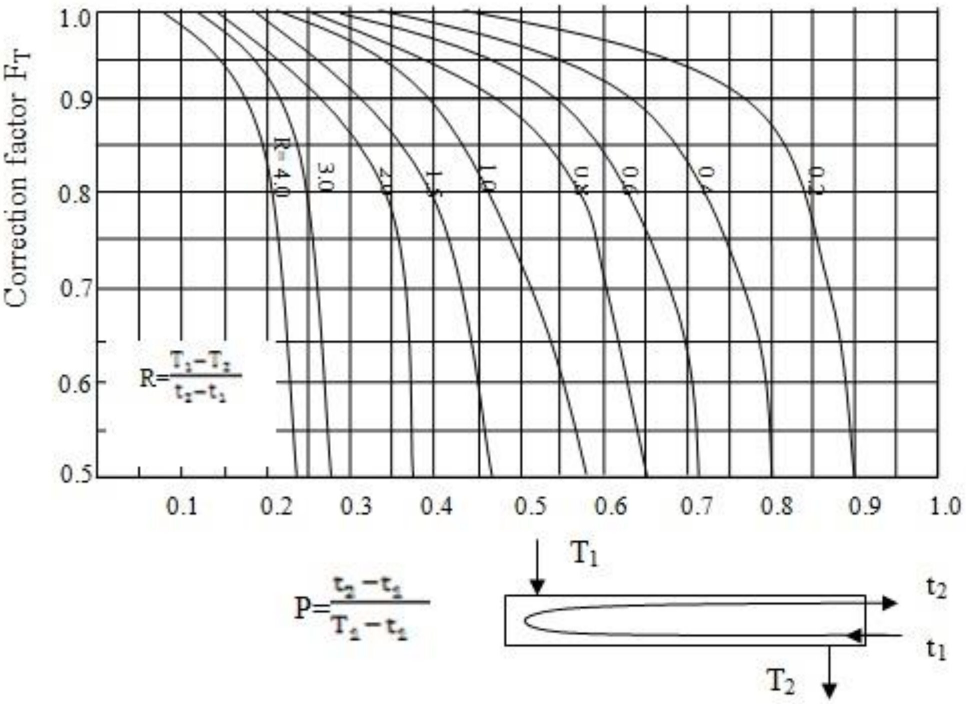


Fig. 8.12: F_T plot for 1-2 exchanger; t: cold fluid in the tube; T: hot fluid in the shell; 1: inlet; 2: outlet

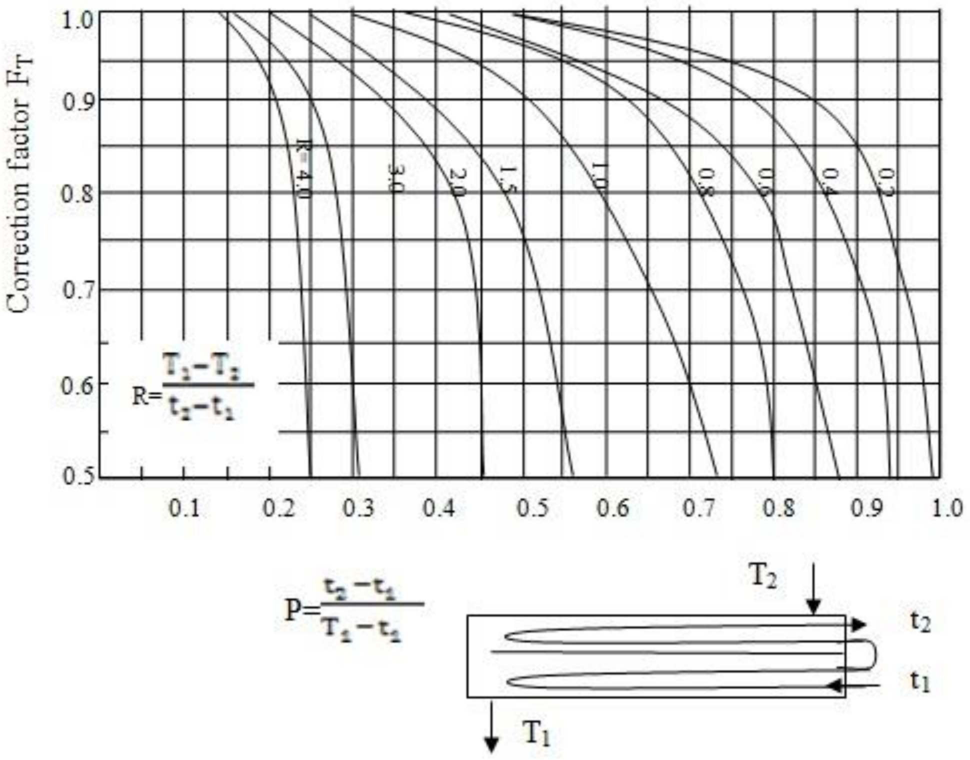


Fig. 8.13: F_T plot for 2-4 exchanger; t: cold fluid in the tube; T: hot fluid in the shell; 1: inlet; 2: outlet

It should be noted that in case of condensation or evaporation the correction factor becomes unity (F_T =1). While designing a heat exchanger, the rule of thumb is that the F_T should not be less than 0.8.

RADIATION

Now, if we observe the heat transfer from the Sun to the earth atmosphere, we can understand that there is no medium exists between the source (the Sun) and the sink (earth atmosphere). However, still the heat transfer takes place, which is entirely a different energy transfer mechanism takes place and it is called thermal radiation.

Thermal radiation is referred when a body is heated or exhibits the loss of energy by radiation. However, more general form "radiation energy" is used to cover all the other forms. The emission of other form of radiant energy may be caused when a body is excited by oscillating electrical current, electronic bombardment, chemical reaction etc. Moreover, when radiation energy strikes a body and is absorbed, it may manifest itself in the form of thermal internal energy, a chemical reaction, an electromotive force, etc. depending on the nature of the incident radiation and the substance of which the body is composed.

In this chapter, we will concentrate on thermal radiation (emission or absorption) that on radiation produced by or while produces thermal excitation of a body.

There are many theories available in literature which explains the transport of energy by radiation. However, a dual theory is generally accepted which enables to explain the radiant energy in the characterisation of a wave motion (electromagnetic wave motion) and discontinuous emission (discrete packets or quanta of energy).

An electromagnetic wave propagates at the speed of light (3×10^8 m/s). It is characterised by its wavelength λ or its frequency ν related by

$$c = \lambda\nu \tag{7.1}$$

Emission of radiation is not continuous, but occurs only in the form of discrete quanta. Each quantum has energy

$$E = h\nu \tag{7.2}$$

where, = 6.6246×10^{-34} J.s, is known as Planck's constant.

Table 7.1 shows the electromagnetic radiation covering the entire spectrum of wavelength

Table 7.1: Electromagnetic radiation for entire spectrum of wavelength

Type	**Band of wavelength (μm)**
Cosmic rays	upto 4×10^{-7}
Gamma rays	4×10^{-7} to 1.4×10^{-4}
X-rays	1×10^{-5} to 2×10^{-2}
Ultraviolet rays	5×10^{-3} to 3.9×10^{-1}
Visible light	$3.9\times10^{-1}$to 7.8×10^{-1}
Infrared rays	7.8×10^{-1} to 1×10^{3}
Thermal radiation	1×10^{-1} to 1×10^{2}
Microwave, radar, radio waves	1×10^{3} to 5×10^{10}

It is to be noted that the above band is in approximate values and do not have any sharp boundary.

7.1 Basic definition pertaining to radiation

Before we further study about the radiation it would be better to get familiarised with the basic terminology and properties of the radiant energy and how to characterise it.

As observed in the table 7.1 that the thermal radiation is defined between wavelength of about 1×10^{-1} and 1×10^{2} μm of the electromagnetic radiation. If the thermal radiation is emitted by a surface, which is divided into its spectrum over the wavelength band, it would be found that the radiation is not equally distributed over all wavelength. Similarly, radiation incident on a system, reflected by a system, absorbed by a system, etc. may be wavelength dependent. The dependence on the wavelength is generally different from case to case, system to system, etc. The wavelength dependency of any radiative quantity or surface property will be referred to as a spectral dependency. The radiation quantity may be monochromatic (applicable at a single wavelength) or total (applicable at entire thermal radiation spectrum). It is to be noted that radiation quantity may be dependent on the direction and wavelength both but we will not consider any directional dependency. This chapter will not consider directional effect and the emissive power will always used to be (hemispherical) summed overall direction in the hemisphere above the surface.

7.1.1 Emissive power

It is the emitted thermal radiation leaving a system per unit time, per unit area of surface. The total emissive power of a surface is all the emitted energy, summed over all the direction and all wavelengths, and is usually denoted as E. The total emissive power is found to be dependent upon the temperature of the emitting surface, the subsystem which this system is composed, and the nature of the surface structure or texture.

The monochromatic emissive power E_λ, is defined as the rate, per unit area, at which the surface emits thermal radiation at a particular wavelength λ. Thus the total and monochromatic hemispherical emissive power are related by

$$E = \int_0^\infty E_\lambda d\lambda \tag{7.3}$$

and the functional dependency of E_λ on λ must be known to evaluate E.

7.1.2 Radiosity

It is the term used to indicate all the radiation leaving a surface, per unit time and unit area.

$$J = \int_0^\infty J_\lambda \, d\lambda \tag{7.4}$$

where, J and J_λ are the total and monochromatic radiosity. The radiosity includes reflected energy as well as original emission whereas emissive power consists of only original emission leaving the system. The emissive power does not include any energy leaving a system that is the result of the reflection of any incident radiation.

7.1.3 Irradiation

It is the term used to denote the rate, per unit area, at which thermal radiation is incident upon a surface (from all the directions). The irradiative incident upon a surface is the result of emission and reflection from other surfaces and may thus be spectrally dependent.

$$G = \int_0^\infty G_\lambda d\lambda \tag{7.5}$$

where, G and G_λ are the total and monochromatic irradiation. Reflection from a surface may be of two types specular or diffusive as shown in fig.7.1.

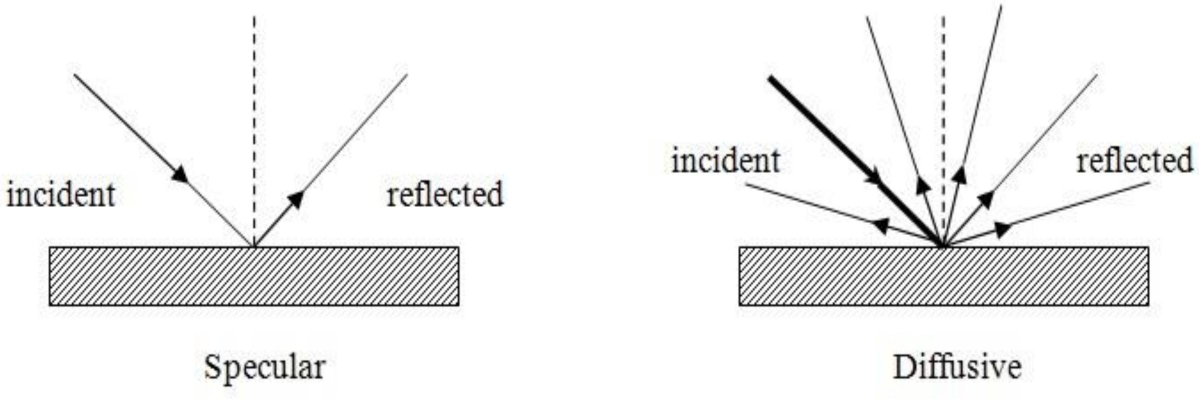


Fig. 7.1: (a) Specular, and (b) diffusive radiation

Thus,

$$J = E + \rho G \tag{7.6}$$

7.1.4 Absorptivity, reflectivity, and transmitting

The emissive power, radiosity, and irradiation of a surface are inter-related by the reflective, absorptive, and transmissive properties of the system.
When thermal radiation is incident on a surface, a part of the radiation may be reflected by the surface, a part may be absorbed by the surface and a part may be transmitted through the surface as shown in fig.7.2. These fractions of reflected, absorbed, and transmitted energy are interpreted as system properties called reflectivity, absorptivity, and transmissivity, respectively.

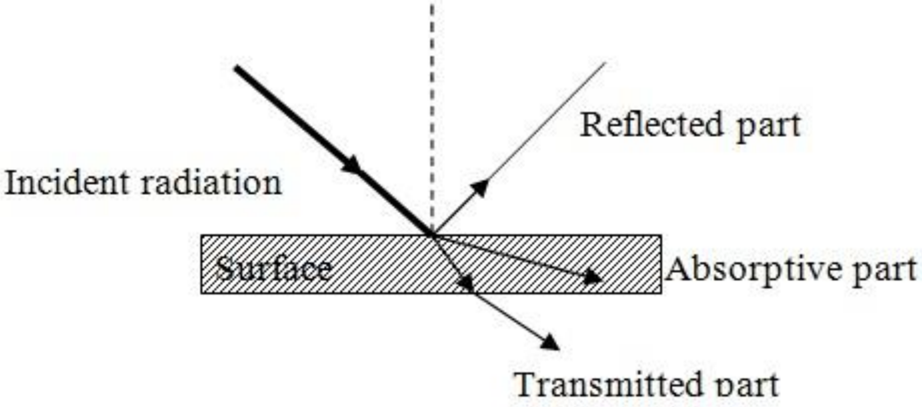


Fig. 7.2: Reflection, absorption and transmitted energy

Thus using energy conservation,

$$\rho + \alpha + \tau = 1 \tag{7.7}$$

$$\rho_\lambda + \alpha_\lambda + \tau_\lambda = 1 \tag{7.7}$$

where, ρ , α and τ are total reflectivity, total absorptivity, and total transmissivity. The subscript λ indicates the monochromatic property.

In general the monochromatic and total surface properties are dependent on the system composition, its roughness, and on its temperature.

Monochromatic properties are dependent on the wavelength of the incident radiation, and the total properties are dependent on the spectral distribution of the incident energy.

Most gases have high transmissivity, i.e. $\tau \approx 1$ and $\rho = \alpha = 0$ (like air at atmospheric pressure). However, some other gases (water vapour, CO_2 etc.) may be highly absorptive to thermal radiation, at least at certain wavelength.

Most solids encountered in engineering practice are opaque to thermal radiation $(\tau \approx 0)$. Thus for thermally opaque solid surfaces,

$$\rho + \alpha = 1 \tag{7.6}$$

Another important property of the surface of a substance is its ability to emit radiation. Emission and radiation have different concept. Reflection may occur only when the surface receives radiation whereas emission always occurs if the temperature of the surface is above the absolute zero. Emissivity of the surface is a measure of how good it is an emitter.

7.2 Blackbody radiation

In order to evaluate the radiation characteristics and properties of a real surface it is useful to define an ideal surface such as the perfect blackbody. The perfect blackbody is defined as one which absorbs all incident radiation regardless of the spectral distribution or directional characteristic of the incident radiation.

$$\alpha = \alpha_\lambda = 1$$

$$\rho = \rho_\lambda = 0$$

A blackbody is black because it does not reflect any radiation. The only radiation leaving a blackbody surface is original emission since a blackbody absorbs all incident radiation. The emissive power of a blackbody is represented by , and depends on the surface temperature only.

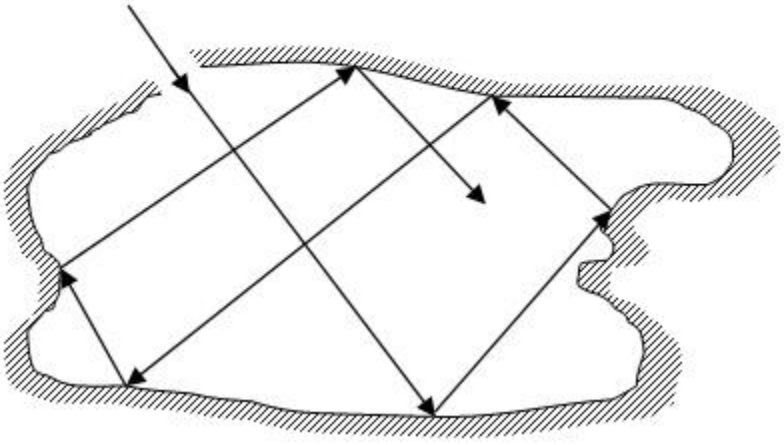


Fig. 7.3: Example of a near perfect blackbody

It is possible to produce a near perfect blackbody as shown in fig.7.3.

Figure 7.2 shows a cavity with a small opening. The body is at isothermal state, where a ray of incident radiation enters through the opening will undergo a number of internal reflections. A portion of the radiation absorbed at each internal reflection and a very little of the incident beam ever find the way out through the small hole. Thus, the radiation found to be evacuating from the hole will appear to that coming from a nearly perfect blackbody.

7.2.1 Planck's law

A surface emits radiation of different wavelengths at a given temperature (theoretically zero to infinite wavelengths). At a fixed wavelength, the surface radiates more energy as the temperature increases. Monochromatic emissive power of a blackbody is given by eq.7.10.

$$E_{b,\lambda} = \frac{2\pi h c^2 \lambda^{-5}}{\exp\left(\frac{hc}{\lambda kT}\right) - 1} \tag{7.7}$$

where, h = 6.6256 X 10^{-34} JS; Planck's constant

c = 3 X 10^{8} m/s; speed of light

T = absolute temperature of the blackbody

λ = wavelenght of the monochromatic radiation emitted

k = Boltzmann constant

Equation 7.10 is known as Planck's law. Figure 7.4 shows the representative plot for Planck's distribution.

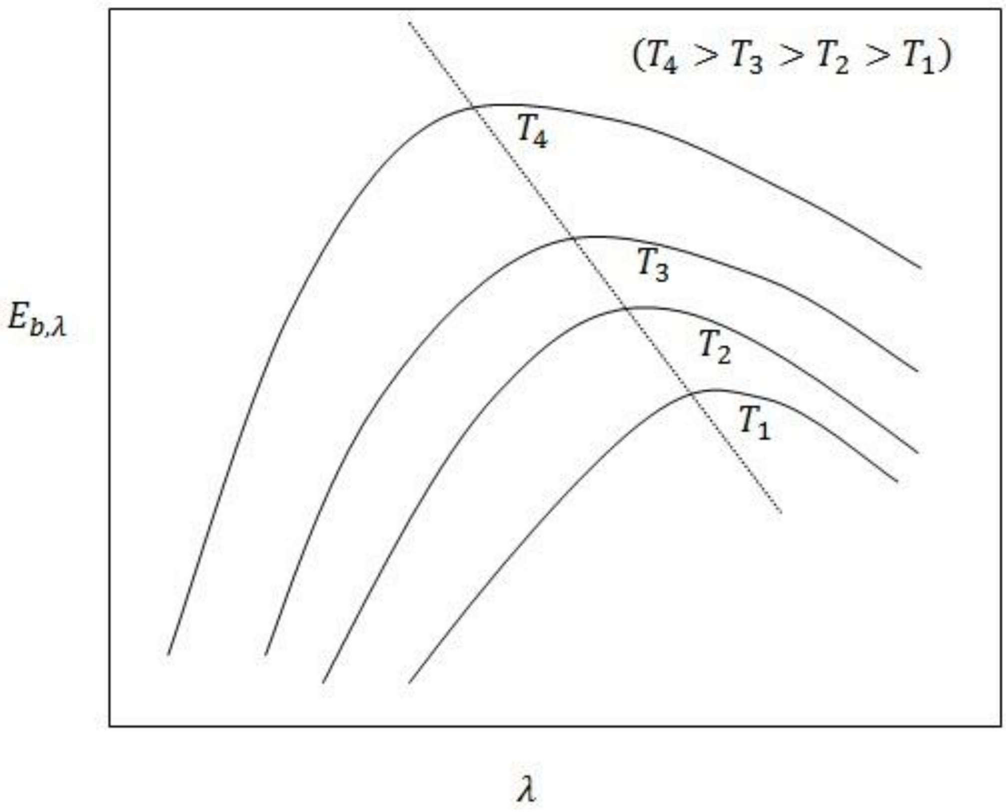


Fig. 7.4: Representative plot for Planck's distribution

7.2.2 Wien's law

Figure 7.4 shows that as the temperature increases the peaks of the curve also increases and it shift towards the shorter wavelength. It can be easily found out that the wavelength corresponding to the peak of the plot (λ_{max}) is inversely proportional to the temperature of the blackbody (Wein's law) as shown in eq. 7.11.

$$\lambda_{max} T = 2898 \qquad (7.11)$$

Now with the Wien's law or Wien's displacement law, it can be understood if we heat a body, initially the emitted radiation does not have any colour. As the temperature rises the λ of the radiation reach the visible spectrum and we can able to see the red colour being height λ (for red colour). Further increase in temperature shows the white colour indicating all the colours in the light.

7.2.3 The Stefan-Boltzmann law for blackbody

Josef Stefan based on experimental facts suggested that the total emissive power of a blackbody is proportional to the fourth power of the absolute temperature. Later, Ludwig Boltzmann derived the same using classical thermodynamics. Thus the eq. 7.12 is known as Stefan-Boltzmann law,

$$E_b = \int_0^\infty E_{b\lambda}(\lambda, T)\, d\lambda$$

$$E_b = \sigma T^4 \qquad (7.12)$$

where, E_b is the emissive power of a blackbody, T is absolute temperature, and σ (= 5.67 X 10^{-8}W/m^2/K^4) is the Stefan-Boltzmann constant.

The Stefan-Boltzmann law for the emissive power gives the total energy emitted by a blackbody defined by eq.7.3.

7.2.4 Special characteristic of blackbody radiation

It has been shown that the irradiation field in an isothermal cavity is equal to E_b. Moreover, the irradiation was same for all planes of any orientation within the cavity. It may then be shown that the intensity of the blackbody radiation, I_b, is uniform. Thus, blackbody radiation is defined as,

$$E_b = \pi I_b \qquad (7.13)$$

where, $I_b = \int_0^\infty I_{b\lambda} d\lambda$ is the total intensity of the radiation and is called the spectral radiation intensity of the blackbody.

7.2.5 Kirchhoff's law

Consider an enclosure as shown in fig.7.2 and a body is placed inside the enclosure. The radiant heat flux (q) is incident onto the body and allowed to come into temperature equilibrium. The rate of energy absorbed at equilibrium by the body must be equal to the energy emitted.

$$EA = \alpha q A$$

$$E = \alpha q \qquad (7.14)$$

where, E is the emissive power of the body, α is absorptivity of the of the body at equilibrium temperature, and A is the area of the body.

Now consider the body is replaced by a blackbody i.e. $E \rightarrow E_b$ and $\alpha = 1$, the equation 7.14 becomes

$$E_b = q \qquad (7.15)$$

Dividing eq. 7.14 by eq.7.15,

$$\frac{E}{E_b} = \alpha \tag{7.16}$$

At this point we may define emissivity, which is a measure of how good the body is an emitter as compared to blackbody. Thus the emissivity can be written as the ratio of the emissive power to that of a blackbody,

$$\frac{E}{E_b} = \epsilon \tag{7.17}$$

On comparing eq.7.16 and eq.7.17, we get

$$\epsilon = \alpha \tag{7.18}$$

Equation 7.18 is the Kirchhoff's law, which states that the emissivity of a body which is in thermal equilibrium with its surrounding is equal to its absorptivity of the body. It should be noted that the source temperature is equal to the temperature of the irradiated surface. However, in practical purposes it is assumed that emissivity and absorptivity of a system are equal even if it is not in thermal equilibrium with the surrounding. The reason being the absorptivity of most real surfaces is relatively insensitive to temperature and wavelength. This particular assumption leads to the concept of grey body. The emissivity is considered to be independent of the wavelength of radiation for grey body.

7.3 Grey body

If grey body is defined as a substance whose monochromatic emissivity and absorptivity are independent of wavelength. A comparative study of grey body and blackbody is shown in the table 7.2.

Table-7.2: Comparison of grey and blackbody

Blackbody	**Grey body**
Ideal body	Ideal body
Emissivity independent of wavelength	Emissivity is independent of wavelength
Absorptivity s independent of wavelength	Absorptivity (α) is independent of wavelength
$\varepsilon = 1$	$\varepsilon < 1$
$\alpha = 1$	$\alpha < 1$

Illustration 7.1

The surface of a blackbody is at 500 K temperature. Obtain the total emissive power, the wavelength of the maximum monochromatic emissive power.

Solution 7.1

Using eq. 7.12, the total emissive power can be calculated,

$$E_b = \sigma T^4$$

where, σ (= 5.67 X 10^{-8} W/m^2/K^4) is the Stefan-Boltzmann constant. Thus at 500 K,

$$E_b = (5.67 \text{ X } 10^{-8})(5000^4) \; W/m^2$$

$$E_b = 354.75 \; W/m^2$$

The wavelength of the maximum monochromatic emissive power can be obtained from the Wien’s law (eq. 7.11),

$$\lambda_{max} T = 2898$$

$$\lambda_{max} = \frac{2898}{500} = 5.796 \, \mu m$$

7.4 Radiative heat exchanger between surfaces

Till now we have discussed fundamental aspects of various definitions and laws. Now we will study the heat exchange between two or more surfaces which is of practical importance. The two surfaces which are not in direct contact, exchanges the heat due to radiation phenomena. The factors those determine the rate of heat exchange between two bodies are the temperature of the individual surfaces, their emissivities, as well as how well one surface can see the other surface. The last factor is known as view factor, shape factor, angle factor or configuration factor.

7.4.1 View factor

In this section we would like to find the energy exchange between two black surfaces having area $A_1$and A_2, respectively, and they are at different temperature and have arbitrary shape and orientation with respect to each other. In order to find the radiative heat exchange between the bodies we have to first define the view factor as

F_{12} = fraction of the energy leavings surface 1 which reaches surface 2

F_{21} = fraction of the energy leaving surface 2 which reaches surface 1 or in general,

F_{mn} = fraction of the energy leaving surface m which raches surface n

Thus the energy leaving surface 1 and arriving at surface 2 is $E_{b1}A_1F_{12}$ and the energy leaving surface 2 and arriving at surface 1 is $E_{b2}A_2F_{21}$. All the incident radiation will be absorbed by the blackbody and the net energy exchange will be,

$$Q = E_{b1}A_1F_{12} - E_{b2}A_2F_{21}$$

At thermal equilibrium between the surfaces $Q_{12} = 0$ and $E_{b1} = E_{b2}$, thus

$0 = E_{b1} (A_1F_{12} - A_2F_{21})$

$$A_1F_{12} - A_2F_{21} \tag{7.19}$$

Equation 7.19 is known as reciprocating relation, and it can be applied in general way for any blackbody surfaces.

$$A_iF_{ij} - A_jF_{ji} \tag{7.20}$$

Though the relation is valid for blackbody it may be applied to any surface as long as diffuse radiation is involved.

7.4.1.1 Relation between view factors

In this section we will develop some useful relation of view factor considering fig. 7.5

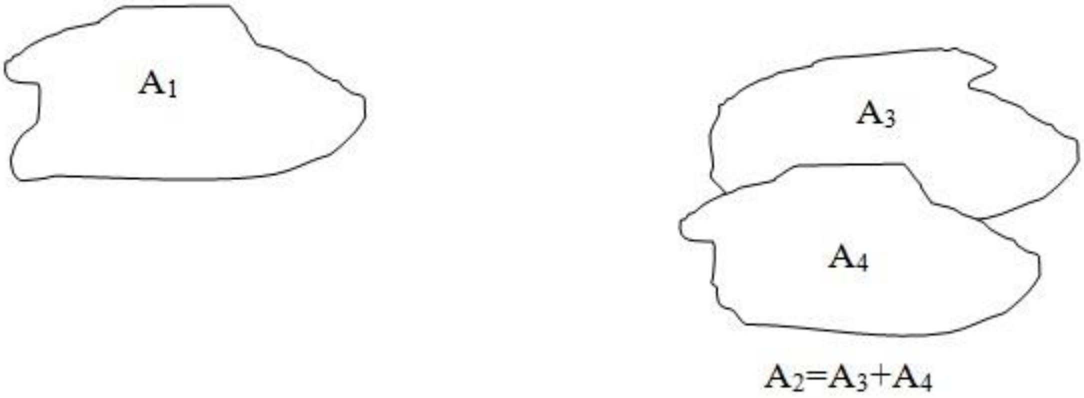


Fig. 7.5: Exchange of energy between area A1 and A2 (A is area of blackbody)

View factor for radiation from A_1 to the combined area A_2,

$$F_{12} = F_{13} + F_{14} \tag{7.21}$$

and using the reciprocating relations for surface 1 and 4,

$$A_1F_{14} = A_4F_{41} \tag{7.22}$$

Using eq. 7.21 and 7.22,

$$F_{41} = \frac{A_1}{A_4} F_{14}$$

$$F_{41} = \frac{A_1}{A_4} (F_{12} - F_{13})$$

Thus the unknown view factor F_{14} can be estimated if the view factors F_{12} and F_{13}, as well as their areas are (A_1, A_2) known.

Now, consider a flat plate (for eg.) which is emitting the radiation, it can be understood that the radiation of the flat plat cannot fall on its own surface (partly or fully). Such kind or surfaces are termed as “not able to see itself”. In such situations,

$$F_{11} = F_{22} = F_{33} = F_{44} = 0$$

However, if the surface can see itself like concave curved surfaces, which may thus see themselves and then the shape factor will not be zero in those cases.

Another property of the shape factor is that when the surface is enclosed, then the following relation holds,

$$\sum_{j=1}^{n} F_{ij} = 1 \tag{7.23}$$

where, F_{ij} is the fraction of the total energy leaving surface i which arrives at surface j.

In case of N-walled enclosure, some of the view factors may be evaluated from the knowledge of the rest and the total N^2 view factors may be represented in square matrix form shown below,

$$\begin{bmatrix} F_{11} & F_{12} & \cdots F_{1N} \\ F_{21} & F_{22} & \ldots F_{2N} \\ \cdots & \cdots & \cdots\cdots \\ F_{N1} & F_{N2} & \cdots F_{NN} \end{bmatrix}$$

Illustration 7.2

A pipe having 10 cm of diameter is carrying saturated steam at 8 bar of absolute pressure. The pipe runs through a room. The wall of the room is at 300 °K. A portion around 1 m of the pipe insulation is damaged and exposed to the room atmosphere. Calculate the net rate of heat loss from the pipe by radiation.

Solution 7.2

The emissivity of the pipe surface is not given so it may be considered black. Moreover, since the room may be big compared to the surface area of the pipe, the room may also be considered to be a blackbody.
We can write $F_{11} + F_{12} = 1$.
The value of $F_{11} = 0$, as the pipe cannot see itself.
Thus F_{12}, the view factor (1-pipe, 2-room) will be 1.
The net rate heat loss due to radiation,

$$Q_{12} = A_{pipe}\epsilon_{pIpe}F_{12}\sigma\,(T_{pipe}^{4} - T_{room}^{4})$$

T_{pipe} can be obtained by the temperature of the steam at the prevailing pressure with the help of steam table = 450 K.

σ (= 5.67×10^{-8} W/m^2/K^4)

On putting the value,

$Q_{12} = \{\pi(0.1)(1)\}(1)(1)(5.67 \times 10^{-8})\{450^4 - 300^4\}$

$Q_{12} = 586\ W$

7.5 Heat exchange between non blackbodies

Evaluation of radiative heat transfer between black surfaces is relatively easy because in case of blackbody all the radiant energy which strikes the surface is absorbed. However, finding view factor is slightly complex, but once it can be done, finding heat exchange between the black bodies is quite easy.

When non blackbodies are involved the heat transfer process becomes very complex because all the energy striking on to the surface does not get absorbed. A part of this striking energy reflected back to another heat transfer surface, and part may be reflected out from the system entirely. Now, one can imagine that this radiant energy can be reflected back and forth between the heat transfer surfaces many times.

In this section, we will assume that all surfaces are in the analysis are diffuse and uniform in temperature and that the reflective and emissive properties are constant over all surfaces.

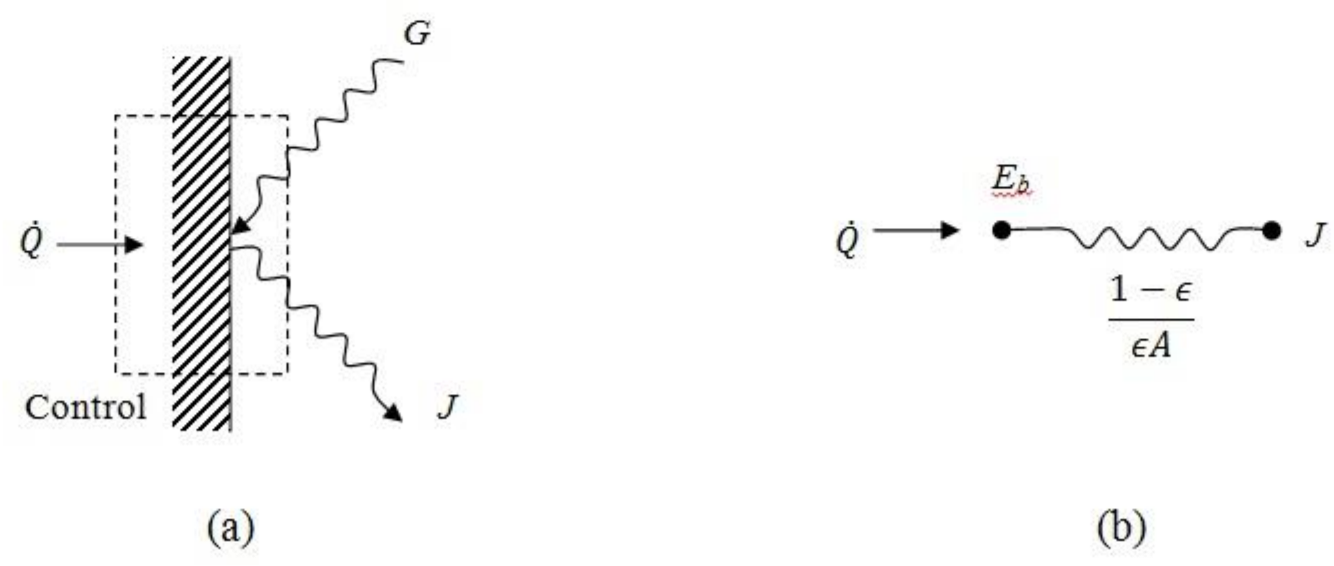


Fig. 7.6: (a) Surface energy balance for opaque surface (b) equivalent electrical circuit

It is also assumed that the radiosity and irradiation are uniform over each surface. As we have already discussed that the radiosity is the sum of the energy emitted and the energy reflected when no energy is transmitted (as opaque body), or

$$J = \epsilon E_b + \rho G \tag{7.24}$$

where, ϵ is the emissivity and E_b is the blackbody emissive power. Because the transmissivity is zero due to opaque surface and absorptivity of the body (grey) will be equal to its emissivity by Kirchhoff's law.

$$\rho = 1 - \alpha = 1 - \epsilon$$

Thus, eq.7.24 becomes

$$J = \epsilon E_b + (1 - \epsilon)G \tag{7.25}$$

The net energy leaving the surface is the difference between the radiosity and the irradiance (fig.7.6a),

$$\frac{\dot{Q}}{A} = J - G = \epsilon E_b + (1-\epsilon)G - G$$

$$\frac{\dot{Q}}{A} = \frac{\epsilon A}{1-\epsilon}(E_b - J)$$

$$\dot{Q} = \frac{E_b - J}{(1-\epsilon)/\epsilon A} \tag{7.26}$$

The eq.7.26 can be analogous to the electrical circuit as shown in fig.7.6(b). The numerator of the eq.7.26 is equivalent to the potential difference, denominator is equivalent to the surface resistance to radiative heat, and left part is equivalent to the current in the circuit.

In the above discussion we have considered only one surface. Now we will analyse the exchange of radiant energy by two surfaces, A_1 and A_2, as shown in the fig.7.7a.

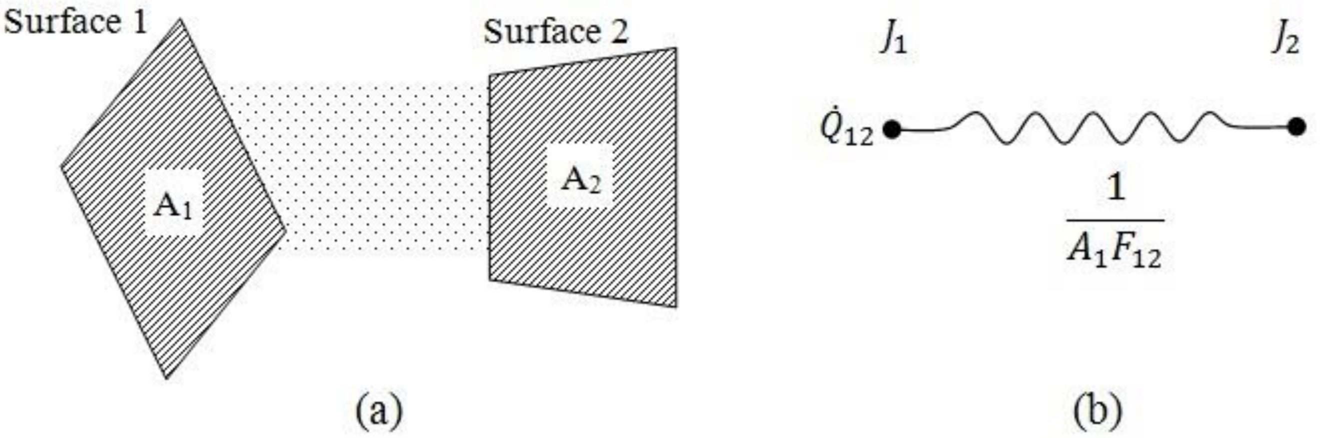


Fig. 7.7: (a) Energy exchange between two surfaces, (b) equivalent circuit diagram

The radiation which leaves surface 1, the amount that reaches surface 2 is

$$J_1A_1F_{12}$$

Similarly, the radiation which leaves system 2, the amount that reaches surface 1 is

$$J_2A_2F_{21}$$

The net energy transfer between the surfaces,

$$\dot{Q}_{12} = J_1A_1F_{12} - J_2A_2F_{21}$$

Reciprocity theorem states that

$$A_1F_{12} = A_2F_{21}$$

$$\Rightarrow \dot{Q}_{12} = (J_1 - J_2)A_1F_{12} = (J_1 - J_2)A_2F_{21}$$

$$\Rightarrow \dot{Q}_{12} = \frac{(J_1 - J_2)}{1/\,A_1F_{12}} \qquad (7.27)$$

It also resembles an electrical circuit shown in fig.7.7b. The difference between eq.7.26 and 7.27 is that in eq.7.27 the denominator term is space resistance instead of surface resistance.

Now, to know, the net energy exchange between the two surfaces we need to add both the surface resistances along with the overall potential as shown in the fig.7.8. Here the surfaces see each other and nothing else.

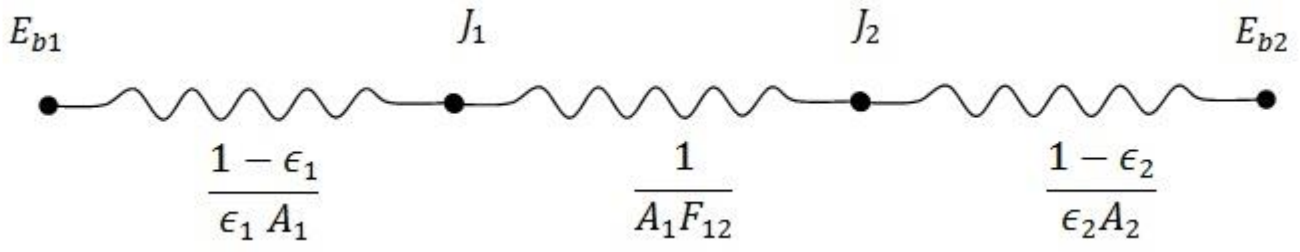


Fig. 7.8: Radiative nature for two surfaces which can see each other nothing else

$$\dot{Q}_{12} = \frac{E_{b1} - E_{b2}}{\frac{1-\epsilon_1}{\epsilon_1 A_1} + \frac{1}{A_1F_{12}} + \frac{1-\epsilon_2}{\epsilon_2 A_2}} \qquad (7.28)$$

7.6 Radiation shield

Till now we have discussed about the radiative heat transfer from one surface to another without any interfering surface in between. Here we will discuss about an interfering shield in between, which is termed as radiation shield. A radiation shield is a barrier wall of low emissivity placed between two surfaces which reduce the radiation between the bodies. In fact, the radiation shield will put additional resistance to the radiative heat transfer between the surfaces as shown in fig.7.9.

Fig. 7.9: Radiation between two large infinite plates (a) without and (b) with radiation shield

Considering fig.7.9(b) and the system is at steady state, and the surfaces are flat (F_{ij} because each plate is in full view of the other). Moreover, the surface are large enough and $\frac{A_1}{A_2} \approx 1$ may be considered and the equivalent blackbody radiation energy may be written as $E_b = \sigma T^4$.

Thus, eq. 7.28 becomes

$$\left.\frac{\dot{Q}}{A}\right|_{net} = \left.\frac{\dot{Q}_{13}}{A_1}\right|_{net} = \left.\frac{\dot{Q}_{32}}{A_3}\right|_{net} = \frac{\sigma\left(T_1^4 - T_3^4\right)}{\frac{1}{\epsilon_1}+\frac{1}{\epsilon_3}-1} = \frac{\sigma\left(T_3^4 - T_2^4\right)}{\frac{1}{\epsilon_3}+\frac{1}{\epsilon_2}-1} \qquad (7.29)$$

In order to have a feel of the role of the radiation shield, consider that the emissivities of all the three surfaces are equal.

$$\epsilon_1 = \epsilon_2 = \epsilon_3 = \epsilon$$

Then it can be seen that the heat flux is just one half of that which would be experienced if there were no shield present.
In similar line we can deduce that when n-shields are arranged between the two surfaces then,

$$\left(\frac{\dot{Q}}{A}\right)_{net_{with\ shield}} = \frac{1}{n+1}\left(\frac{\dot{Q}}{A}\right)_{without\ shield} \qquad (7.30)$$

7.7 Electrical network for radiation through absorbing and transmitting medium

The previous discussions were based on the consideration that the heat transfer surfaces were separated by a completely transparent medium. However, in real situations the heat transfer medium absorbs as well as transmits. The examples of such medium are glass, plastic film, and various gases.

Consider two non-transmitting surfaces (same as in fig. 7.8) are separated by a transmitting and absorbing medium. The medium may be considered as a radiation shield which see themselves and others. If we distinguish the transparent medium by *m* and if the medium is non-reflective (say gas) then using Kirchhoff's law,

$$\alpha_m + \tau_m = 1 = \epsilon_m + \tau_m \tag{7.31}$$

The energy leaving surface 1 which is transmitted through the medium and reaches the surface 2 is,

$$J_1 A_1 F_{12} \tau_m$$

and that which leaves surface 2 and arrives at surface 1 is,

$$J_2 A_2 F_{21} \tau_m$$

Therefore, the net exchange in the transmission process is therefore,

$$\dot{Q}_{12} = A_1 F_{12} \tau_m (J_1 - J_2)$$

Using eq. 7.31,

$$\dot{Q}_{12} = \frac{(J_1 - J_2)}{\left(\frac{1}{A_1 F_{12}(1-\epsilon_m)}\right)}$$

Thus the equivalent circuit diagram is shown in fig. 7.9

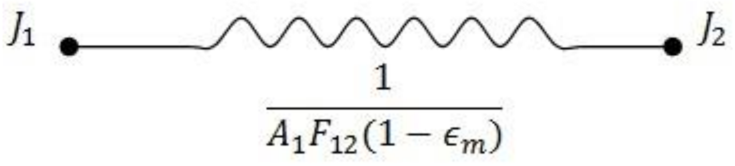


Fig. 7.9. Equivalent electrical circuit for radiation through gas

7.8 Radiation combined with conduction and convection

In industrial processes, in general, the heat transfer at higher temperature has significant portion of radiation along with conduction and convection. For example, a heated surface is shown in the fig. 7.10 with all the three mechanism of heat transfer.

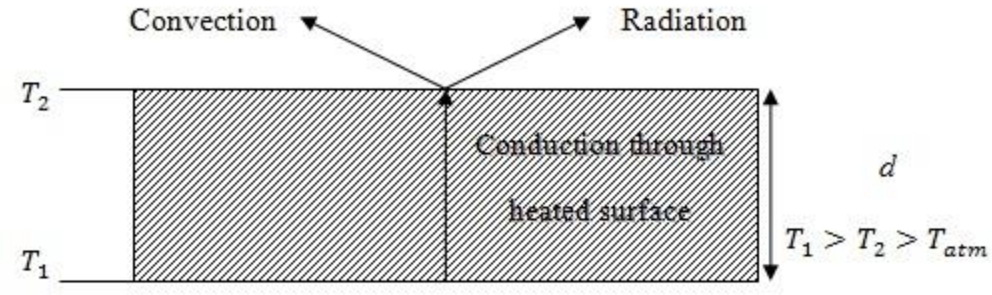


Fig. 7.11: Radiation combined with conduction and convection

At steady state

Heat flux by conduction = heat flux by convention + heat flux by radiation

$$\frac{K}{d}(T_1 - T_2) = h(T_2 - T_{atm}) + \epsilon\sigma(T_2^4 - T_{atm}^4)$$

where, h is the heat transfer coefficient at the surface in contact (outer surface) with atmosphere due to natural and forced convection combined together, ϵ is the emissivity of the outer surface, andT_{atm} is the atmospheric temperature.

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DEPARTMENT OF CHEMICAL ENGINEERING

UNIT – 5 HEAT TRANSFER OPERATIONS -SCHA 1311

UNIT 5

HEAT TRANSFER OPERATIONS

Evaporators- natural circulation, forced-circulation and agitated-film evaporators. Methods of operation of evaporators-single-effect and multiple-effect evaporators, Evaporation of Biological materials- fruit juices, sugar solution and paper-pulp waste liquors.

Definition:

Theoretically, evaporation means simply vaporization from the surface of the liquid.

N.B. Thus no boiling occurs and the rate of vaporization from the depends on the diffusion of vapor through the boundary layers above the liquid. The partial pressure of vapor is the driving force here. In practice rate of heat transfer by this process is very slow.

Practically, evaporation is defined as the removal of liquid from a solution by boiling the liquor in a suitable vessel and withdrawing the vapor, leaving a concentrated liquid residue.

Objective of evaporation:

To make a solution more concentrated. Generally extracts are concentrated in this way.

Factors affecting evaporation:

(i) Temperature:

Heat is necessary to provide the latent heat of vaporization, and in general, the rate of evaporation is controlled by the rate of heat transfer. Rate of heat transfer depends on the temperature gradient.

Many pharmaceutical agents are thermolabile. So the temperature that will cause the least possible decomposition should be used.

e.g. Many glycosides and alkaloids are decomposed at temperature below 100^0C.

e.g. Hormones, enzymes and antibiotics are extremely heat sensitive substances. e.g. Malt extract (containing enzyme) is prepared by evaporation under reduced pressure to avoid loss of enzymes.

Some antibiotics are concentrated by freeze-drying.

(ii) Temperature and time of evaporation

Exposure to a relatively high temperature for a short period of time may be less destructive of active principles than a lower temperature with exposure for a longer period.

Film evaporators used a fairly high temperature but the time of exposure is very short.

An evaporating pan involve prolonged heating.

(iii) Temperature and moisture content

Some drug constituents decompose more rapidly in the presence of moisture, especially at a raised temperature (by hydrolysis). Hence, evaporation should be carried out at a low controlled temperature, although the final drying can be performed at higher temperature when little moisture remains.

e.g. Belladonna Dry Extract is an example of this type.

(iv) Type of product required

Evaporating pans or stills will produce liquid or dry products, but film evaporators will yield only liquid products. So a dilute extract can be first concentrated in a film evaporator and then the concentrated extract may be died in an evaporating pan.

(v) Effect of concentration

As the liquor becomes concentrated, the increasing proportion of solids results in elevation of the boiling point of the solution. This leads to a greater risk of damage to thermolabile constituents and reduction of the temperature gradient.

In general concentrated solutions will have increased viscosity, causing thicker boundary layers, and may deposit solids that may build up on the heating surface that reduce heat transfer.

All these problems may be minimized by turbulent flow condition.

EVAPORATORS

Evaporators are classified according to the form of the movement,

(i) Natural circulation evaporators.

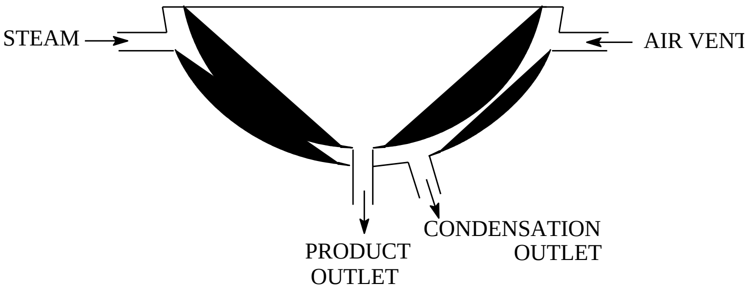
(ii) Forced circulation evaporation

(iii) Film evaporators

(i)Natural Circulation Evaporator

EVAPORATING PAN

Description



In these evaporators the movement of the liquid results from convection currents set up by the heating process.

The apparatus consists of a hemispherical, or shallower pan, constructed from a suitable material such as copper or stainless steel and surrounded by a steam jacket.

The hemispherical shape gives the best surface/ volume ratio for heating, and the largest area for disengagement of vapor.

The pan may have a mounting , permitting it to be tilted to remove the product, but the shallow form makes this arrangement somewhat unstable, and an outlet at the bottom, is common.

Advantages:

(a) It is simple and cheap to construct.

(b) It is easy to use, clean and maintain.

Disadvantages:

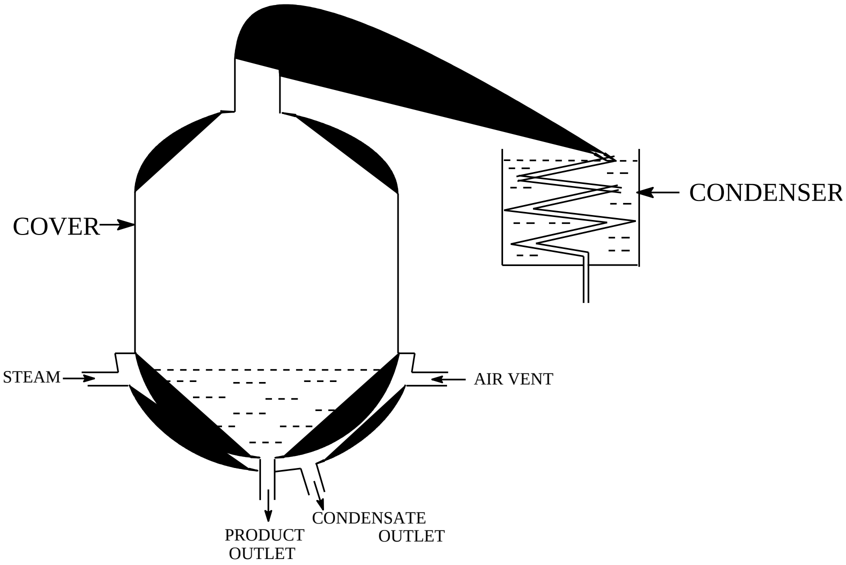
(a) Having only natural circulation, the overall coefficient of heat transfer will be poor and solids are likely to deposit on the surface, leading to decomposition of the product and a further deterioration in heat transfer.
(b) Also many products give rise to foaming.
(c) The total liquor is heated over all the time, which may be unsatisfactory with thermolabile materials.

(d) The heating surface is limited and decreases proportionally as the size of the pan increases.
(e) The pan is open, so the vapor passes to the atmosphere which can lead to saturation of the atmosphere.
(f) Only aqueous liquids can be evaporated in this pans.
(g) Pan evaporation cannot be done under reduced pressure.
(h) Can only be used for thermolabile products.

EVAPORATING STILLS

They are called stills because it is essentially a vessel similar to the evaporating pan, with a cover that connects it to a condenser, so that the liquid is distilled off.

Often a quick release system of clamps which allows the cover to be removed easily for access to the interior of the vessel for cleansing or removal of the product may be used.



Advantages:

(a) Simple construction and easy to clean and maintain.

(b) The vapor is removed by condensation which

- (i) speeds evaporation
- (ii) reduces inconvenience and
- (iii)allows the equipment to be used for solvents other than water e.g. ethanol.

(c) A receiver and vacuum pump can be fitted to the condenser, permitting operation under reduced pressure and, hence, at lower temperature.

Disadvantages:

(a) Natural convection only

(b) All the liquor is heated all the time

(c) The heating surface is limited.

Uses:

(i) Aqueous and other solvents may be evaporated
(ii) Thermolabile materials can be evaporated under reduced pressure.
(iii) Removing the still head it is convenient for evaporating extracts to dryness.

SHORT TUBE EVAPORATOR (Basket type vertical short tube evaporator)

Construction and Principle

Calendria

The lower portion of the evaporator consist of a nest of tubes with the liquor inside and steam outside.

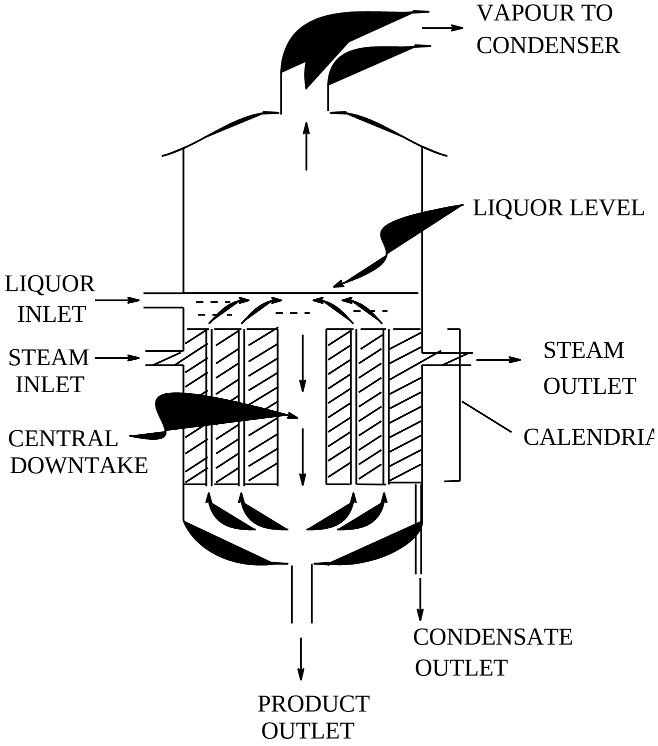
Tube length: 1 – 2 m

Tube diameter: 40 – 80 mm

Diameter of evaporator: 2.5 m

Number of tubes: 1000

This part of the evaporator is called the calendria.



- The liquor is maintained at a level slightly above the top of the tubes, the space above this being left for the disengagement of vapor from the boiling liquor.
- The liquor in the tubes is heated by the steam and begins to boil, when the mixture of liquid and vapor will shoot up the tubes (in a similar manner to that of a liquid that is allowed to boil to vigorously in a test-tube).
- This sets up a circulation, with boiling

liquor rising up the smaller tubes of the calendria and returning down the larger central downtake.

Advantages

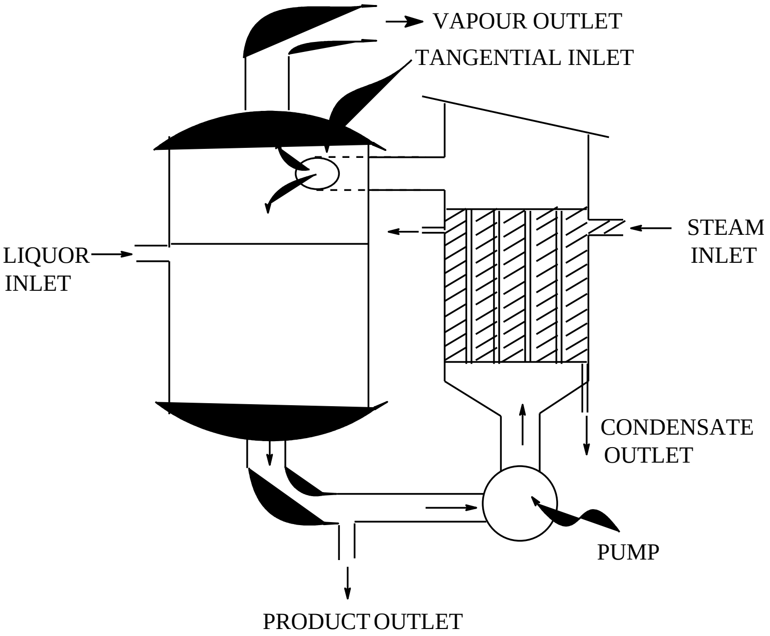
1. Use of tubular calendria increases the heating area, possibly by a factor of 10 to 15 compared to that of an external jacket.
2. The vigorous circulation reduces boundary layers and keeps solids in suspension, so increasing the rate of heat transfer.
3. Condenser and receiver can be attached to run the evaporation under vacuum with nonaqueous solvents.

Disadvantages

1. Since the evaporator is filled to a point above the level of the calendria, a considerable amount of liquid is heated for a long time. The effect of this continual heating can be reduced to some extent by removing concentrated liquor slowly from the outlet at the bottom of the vessel.
2. Complicated design, difficult for cleaning and maintenance.
3. The head (pressure) of the liquor increases pressure at the bottom of the vessel and, in large evaporators where the liquor depth may be of the order of 2 m; this may give rise to a pressure of about 0.25 bar, leading to elevation of the boiling point by 5 to 6^0C.

FORCED CIRCULATION EVAPORATORS

Forced circulation evaporators are natural circulation evaporators with some added form of mechanical agitation. Different forms of forced circulation evaporators can be designed.



- An evaporating pan, in which the contents are agitated by a stirring rod or pole could be described as a forced circulation evaporator.
- A mechanically operated propeller or paddle agitator can be introduced into an evaporating pan or still.
- Propeller or paddle agitator can be introduced into the downtake of a short-tube evaporator.
- A typical forced circulation evaporator can be shown as follows:

Working Principle

- The liquor is circulated by means of the pump and as it is under pressure in the tubes the boiling point is elevated and no boiling takes place.
- As the liquor leaves the tubes and enters the body of the evaporator there is a drop in pressure and vapor flashes off from the superheated liquor.

Advantages

- Rapid liquid movement improves heat transfer, especially with viscous liquids or materials that deposit solids or foam readily.
- The forced circulation overcomes the effect of greater viscosity of liquids when evaporated under reduced pressure.
- Rapid evaporation rate makes this method suitable for thermolabile materials, e.g. it is used in practice for the concentration of insulin and liver extracts.

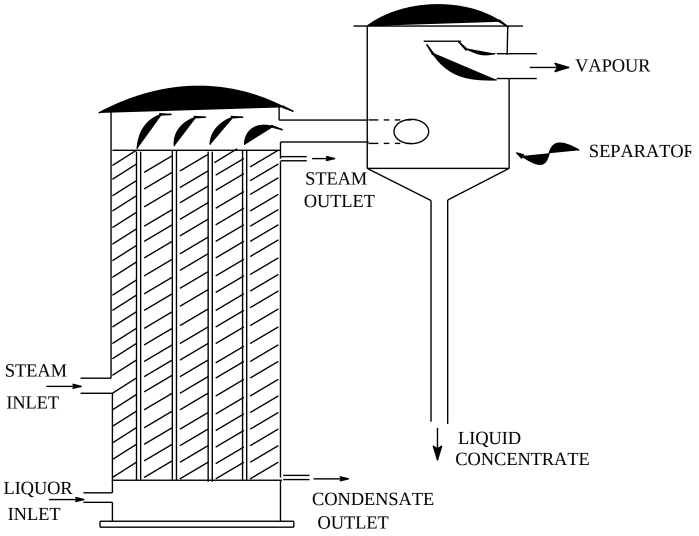
FILM EVAPORATORS

Film evaporators spread the material as a film over the heated surface, and the vapor escapes the film.

Long tube evaporators (Climbing film evaporators)

Construction and working principle

The heating unit consists of steam-jacketed tubes, having a length to diameter ratio of about 140 to 1, so that a large evaporator may have tubes 50 mm in diameter and about 7 m in length.

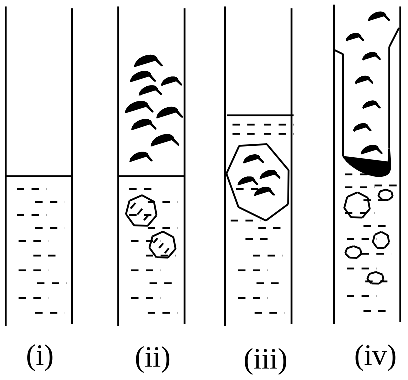


The liquor to be evaporated is introduced into the bottom of the tube, a film of liquid forms on the walls and rises up the tubes, hence it is called <u>climbing film evaporator.</u>

At the upper end, the mixture of vapor and concentrated liquor enters a separator, the vapor passing on to a condenser, and the concentrate to a receiver.

Cold or pre heated liquor is introduced into the tube (i). Heat is transferred to the liquor from the walls and boiling begins, increasing in vigor (ii). Ultimately sufficient vapor has been formed for the smaller bubbles to unite to a large bubble, filling the width of the tube and trapping a 'slug' of liquid above the bubble (iii).

As more vapor is formed, the slug of liquid is blown up the tube, the tube is filled with vapor, while the liquid continues to vaporize rapidly, the vapor escaping up the tube and, because of friction between the vapor and liquid, the film also is dragged up the tube upto a distance of 5 to 6 metres.



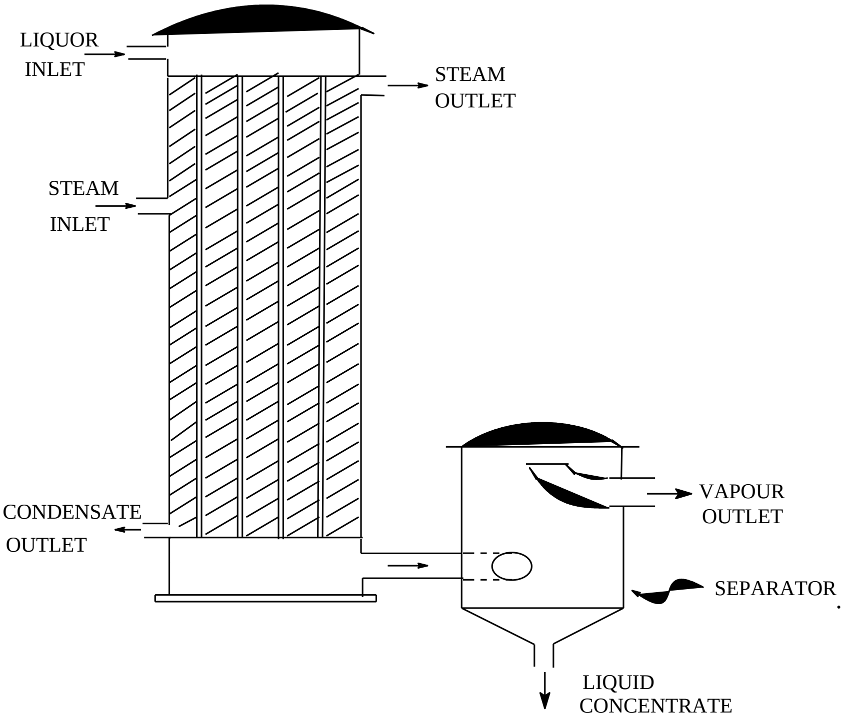
Long tube evaporators (Falling film evaporators)

Construction and working principle

Construction is same as climbing film evaporator but is inverted as shown in the figure.

The liquor to be evaporated is introduced at the top of the evaporator tubes and the liquor comes down due to gravity.

The concentrate and vapor leaves the bottom. They are separated in a chamber where the concentrate is taken out through product outlet out through product outlet and vapor from vapor outlet.



Advantages of long tube evaporator

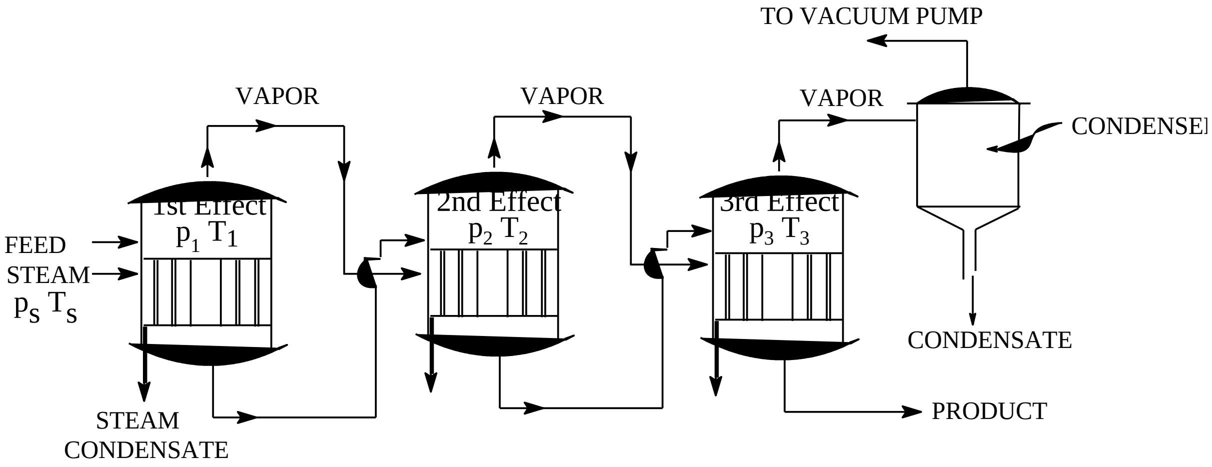
Since the movement of the film is assisted by gravity, more viscous liquid can be handled by falling film evaporator.

(i) Very high film velocity reduces boundary layers to a minimum giving improved heat transfer.
(ii) The use of long narrow tubes provides large surface area for heat transfer.
(iii) Because of increased heat transfer efficiency, a small temperature gradient is necessary with less risk of damage to thermolabile materials.
(iv) Although the tubes are long, they are not submerged, as in the short-tube evaporator; so that there is no elevation of boiling point due to hydrostatic head.

Disadvantages

(i) Expense to manufacture and install the instrument is high.
(ii) Difficult to clean and maintain.
(iii) From the operational point of view the feed rate is critical. if too high, the liquor may be concentrated insufficiently, where as if the feed rate is to low, the film cannot be maintained and dry patches may form on the tube wall.

Multiple effect evaporator



Triple-effect evaporator: p_s, p_1, p_2, p_3 *vapor pressures, Ts, T1, T2, T3 temperatures*

where $p_s > p_1 > p_2 > p_3$.

In a single effect evaporator steam is supplied for heating the liquor. The total heat is not transferred form the steam. So the rest of the heat is wasted. to use that heat efficiently. Connections are made so that the vapor from one effect serves as the heating medium for the next effect.

(i) The dilute feed (liquor) enters the first effect, where it is partly concentrated; it flows to the second effect for additional concentration and then to the third effect for final concentration. This liquor is pumped out of the third effect.

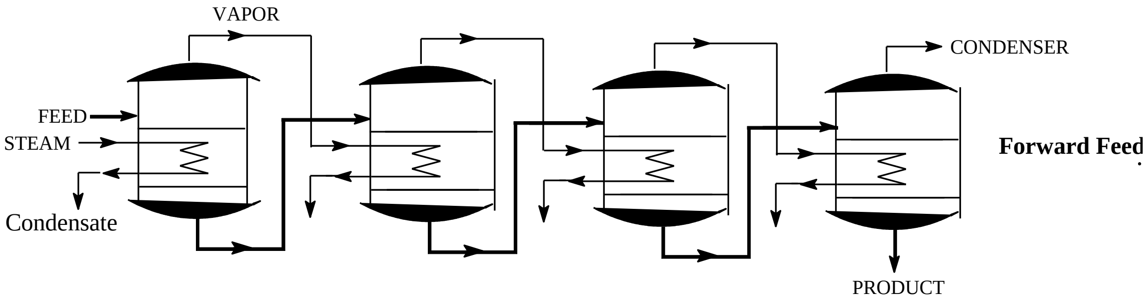
(ii) In the first effect raw steam is fed in which the vapor pressure in the evaporator is the highest, p_1. the second effect has the intermediate vapor pressure; i.e. $p_1>p_2>p_3$. This pressure gradient is maintained by drawing the vapor through a vacuum pump and condensing after the final effect.

(iii) Depending on the lowering of vapor pressure boiling point of the liquids of 2nd and 3rd effect will also be lowered; i.e. $T_1 > T_2 > T_3$.

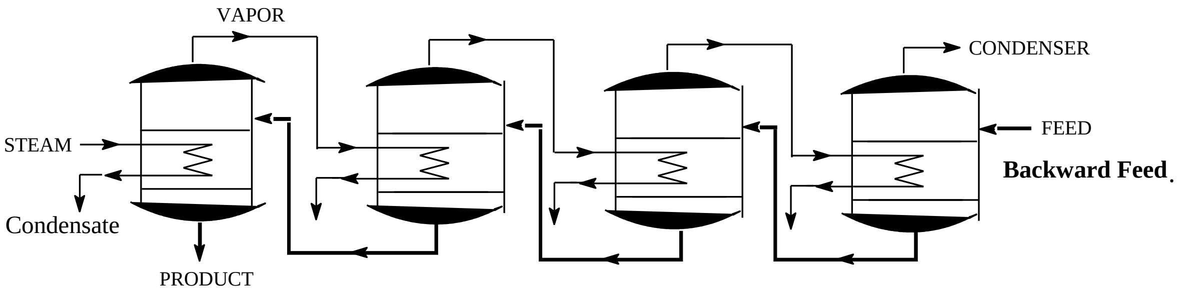
(iv) In the 2nd effect vapor from the 1st effect (T_1) is heating the liquor (having temperature T_2). So there is a temperature gradient ($T_1 – T_2$); consequently the liquor will be heated.

Similar heating will be there in the 3rd effect also.

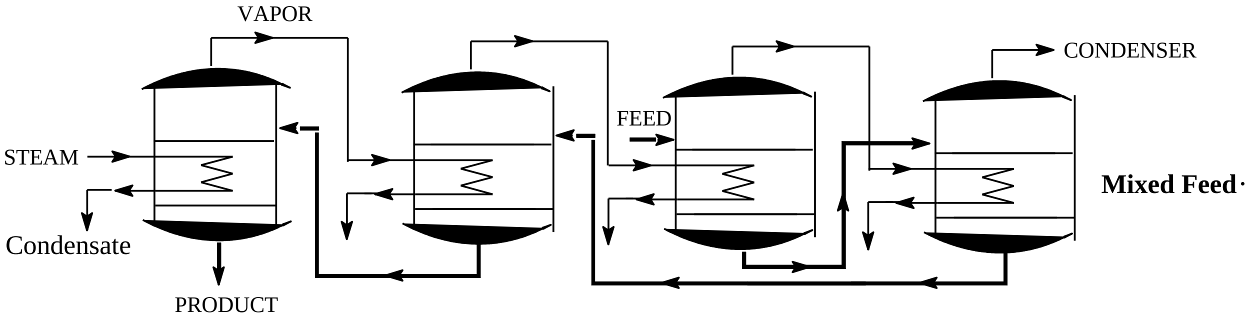
Methods of feeding



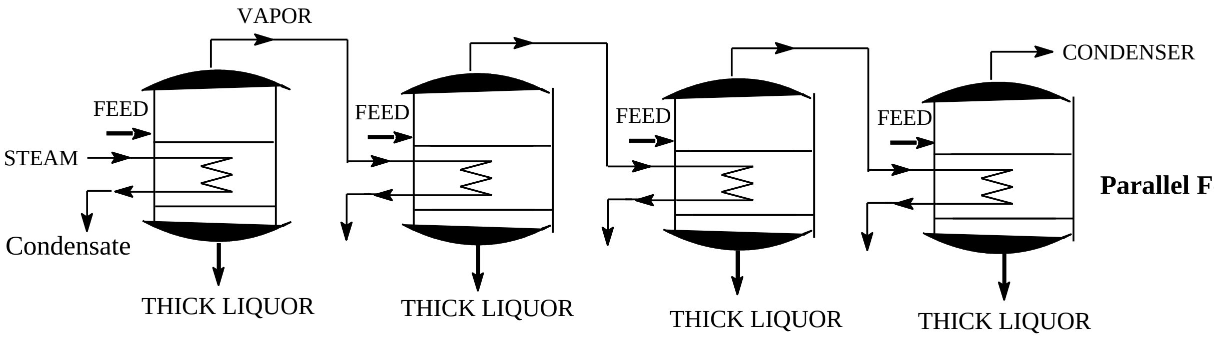
Forward Feed.



Backward Feed.



Mixed Feed·



Parallel Fee

Forward feed

Advantages:

1. Feed moves from high pressure (in effect-2) to low pressure (in effect – 4) chambers, so pumping of liquor is not required.
2. Product is obtained at lowest temperature.
3. This method is suitable for scale-forming liquids because concentrated product is subjected to lowest temperature.

Disadvantages

It is not suitable for cold feed because, the steam input in effect-1 raises the temperature of the feed, and a small amount of heat is supplied as latent heat of vaporization. Therefore, amount of vapor produced will be less than the amount of steam supplied. Lower amount of vapor in effect-1 produces lower amount of vapor in the subsequent effects. Therefore, the overall economy is lower.

Backward feed

In backward-feed the feed enters in the last effect and moves towards first effect (i.e IV→III→II→I).

Advantages

- It is suitable for cold feed, because the heat used for increasing the temperature in IV effect is already used for heating 3 times. This will give more economy.
- The method is suitable for viscous products, because highly concentrated product is at highest temperature, hence lower viscosity (→ higher heat transfer → higher capacity)

Disadvantages

The liquid moves from low-pressure (IV) to high-pressure chambers (III → II → I) pumping is requierd.

Mixed feed method

The feed enters in the intermediate effect, moves forward and then backward to effect-I (III→IV→II→I).

Advantages

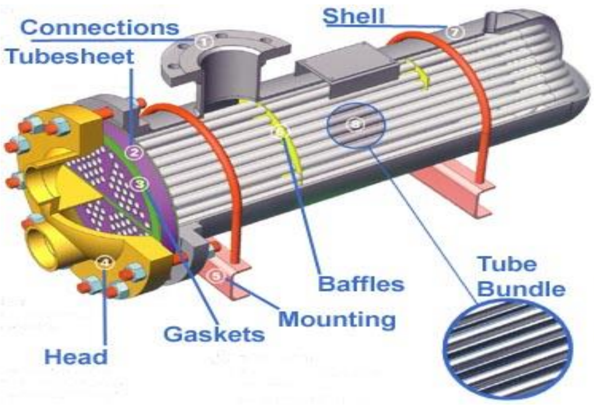
- Liquid moves from high pressure (III) to low pressure (IV), hence no pump is required. Liquid moves from IV→II→I requires pump.
- Product is obtained from highest temperature (I) hence lowest viscosity.

Parallel feed

It is suitable where the feed has to be concentrated slightly.

A **heat exchanger** is a device built for efficient heat transfer from one medium to another, whether the media are separated by a solid wall so that they never mix, or the media are in direct contact. Typical applications involve heating or cooling of a fluid stream of concern and evaporation or condensation of single- or multicomponent fluid streams. In a heat exchanger there is no direct contact between the two fluids. The heat is transferred from the hot fluid to the metal isolating the two fluids and then to the cooler fluid.

In other applications, the objective may be to recover or reject heat, or sterilize, pasteurize, fractionate, distill, concentrate, crystallize, or control a process fluid. They are widely used in Space Heating, Refrigeration, Air Conditioning, Power Plants, Chemical Plants, Petro-chemical Plants, Petroleum Refineries, and Natural Gas Processing. One common example of a heat exchanger is the Radiator in a car.



Different Terminologies of Heat Transfer Equipment

Heat exchanger: both sides single-phase and process streams

Cooler: one stream a process fluid and the other cooling water or air.

Heater: one stream a process fluid and the other a hot utility, such as steam or hot oil.

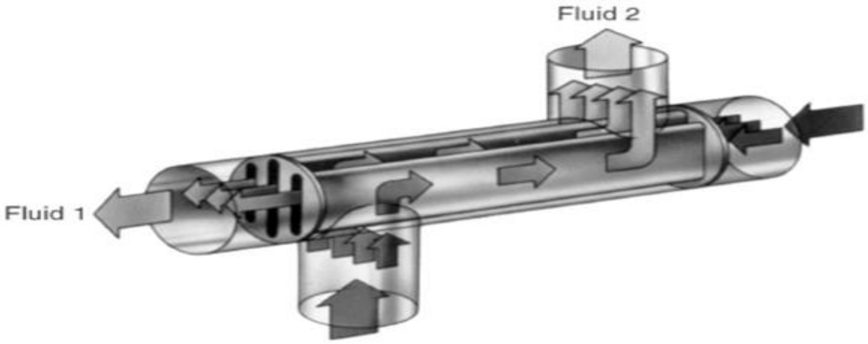
Condenser: one stream a condensing vapor and the other cooling water or air.

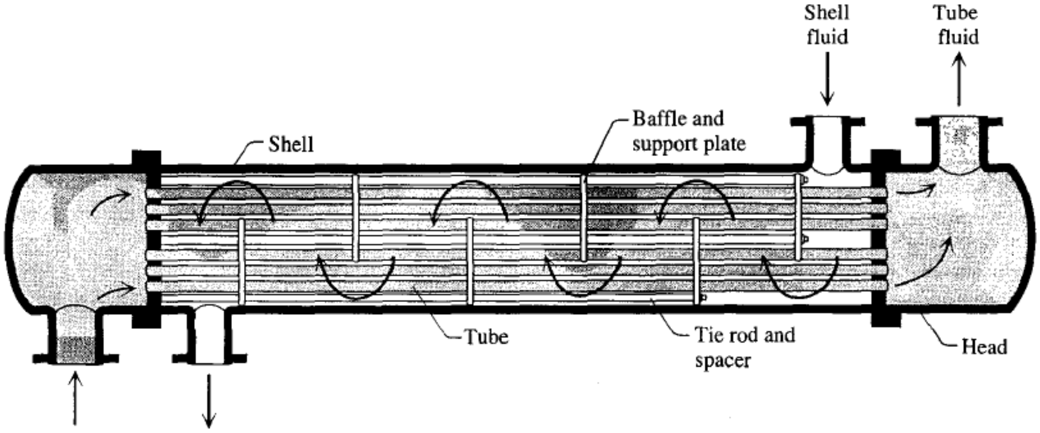
Chiller: one stream a process fluid being condensed at sub-atmospheric temperatures and the other a boiling refrigerant or process stream.

Reboiler: one stream a bottoms stream from a distillation column and the other a hot utility (steam or hot oil) or a process stream.

a. Multiple pass heat exchangers

If fluids pass each other more than once then it applies the principle of multiple pass heat exchangers .Reverse flow are common in these heat exchanger as there are designed with “U”bend tubes. These tubes provide a pathway for back and forth movement of fluid across the length of the heat exchanger.Another way for establishing a multiple pass is to insert baffles on the shell side of the heat exchanger. Baffles directs shell sides the fluid back and forth across the tubes to achieve multiple pass effect. Tubes are sometimes arrayed within the shell in order to shorten the overall exchanger length of the heat exchanger in the opposite direction using the other half of the tube bundle. The exchanger would then be said to have two side tube passes. It has an advantage that the tube side velocities are doubled for the same flow rate thus the heat transfer coefficient increases. Longitudinal baffles are placed in the shell such that the number of shell sides passes can be increased and a shell side coefficient increase consequently. The more complex and costly, the more complex and costly construction and the higher pressure drops for each fluid is off set of multiple pass heat exchanger.



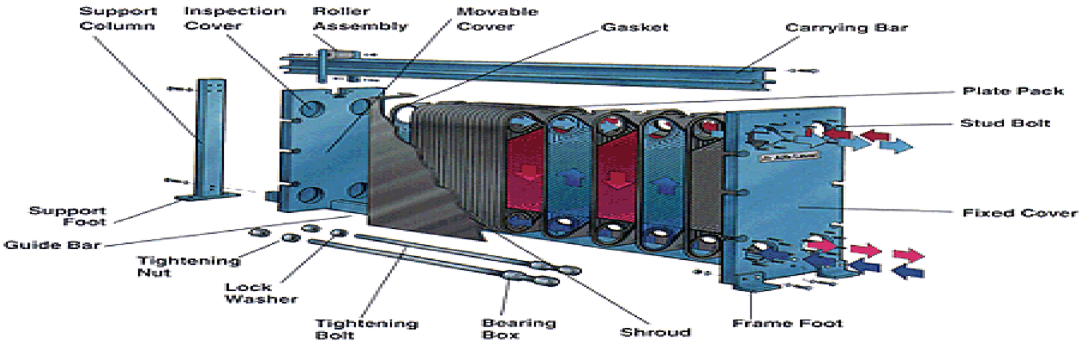


Advantages of Multiple Pass Heat Exchangers

- It has wide ranges of pressure thus pressure can be varied over large drops
- Thermal stresses can be accommodated inexpensively.
- Use of materials is diverse and it take into consideration corrosion and other disruptive concerns .shell and tubes can be made with different material.
- Extended heat transfer surfaces can be used to enhance heat transfer.
- Cleaning and repair are relatively straightforward, because the equipment can be dismantled for this purpose

b. Plate heat exchangers

It’s a type of Heat Exchanger which consists of many corrugated stainless-steel sheets separated by polymer gaskets and clamped into a steel frame.Plate heat exchangers transfer heat by placing thin, corrugated metal sheets side by side and connecting them by gaskets. Flow of the substances to be heated and cooled takes place between alternating sheets allowing heat to transfer through the metal sheets.



The parts and function

The heat exchangers frame is made up of thick steel pressure retaining parts, the fixed cover and the movable cover, that when pulled together with the tightening bolts form the pressure retaining structure for the plates / plate pack. The carrying bar and guide bar act as a carrier and guide to both the plates and the movable cover.**The heat exchanger plates**, which make up the heat transfer surface, are clamped between two plates of steel with the use of the tightening bolts. The heat exchanger construction allows a plate heat exchanger to be easily opened for inspection and cleaning. Each plate has **a gasket** that produces a sealing and channel system through the entire plate pack in which the two heat exchanging media flow in a counter-current direction. The circular portion of the gasket stops the fluid from going across the heat transfer plate and sends it to the next open channel. The remaining portion or field gasket directs the opposing fluid across the heat transfer surface. The **heat transfer plates** with gaskets are arranged in an alternating pattern of left hand flow and right hand flow to direct the fluids in an opposing direction within the heat exchanger. The completed assembly of all the plates and gaskets is called the “plate pack.”

Plate heat exchangers are used because there have high heat transfer area, high heat transfer coefficient. There are compact and have lower floor space requirements. By increasing the number of plates the area of heat exchange can be increased. They are most suitable type heat exchangers for lower flow rates and heat sensitive substances. They can perform multiple duties that can be performed by a single unit. There are classified into gasketed plate heat exchanger, brazed plate heat exchanger, welded plate heat exchangers.

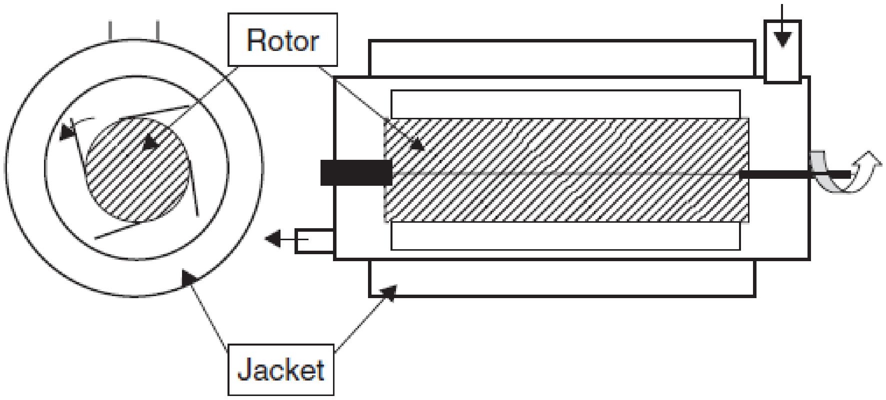
Benefits of plate heat exchangers are lightweight: The PHE unit is lighter in total weight than other types of heat exchangers because of reduced liquid volume space and less surface area for a given application. **High-viscosity applications**: Because the PHE induces turbulence at low fluid velocities, it has practical application for high-viscosity fluids. **Saves space and servicing time**: The PHE fits into an area one-fifth to one-half of that required for a comparable shell and tube heat exchanger. The PHE can be opened for inspection, maintenance. **Lower liquid volume:** Since the gap between the heat transfer plates is relatively small, a PHE contains only low quantities of process fluids. The benefit is reduced cost due to lower volume .**Lower cost:** PHEs are generally more economical than other types of equivalent duty heat exchangers due to the higher thermal efficiency and lower manufacturing costs. **Quick process control:** Owing to the thin channels created between the two adjacent plates, the volume of fluid contained in PHE is small; it quickly reacts to the new process condition and is thereby easier to control**.**

The **limitations of plate heat exchangers are** the maximum allowable working pressure is also limited by the frame strength and plate deformation resistance. Commonly stated limits have been 300°F (149°C) and 300 psi .Because of the narrow gap between the plates, high liquid rates will involve excessive pressure drops, thus limiting the capacity. Large differences in fluid flow rates of two streams cannot be handled. The gaskets cannot handle corrosive or aggressive media. Gaskets always increase the leakage risk. The standard PHEs cannot handle particulates that are larger than 0.5 mm.

In conclusion Plate heat exchangers are available in a wide variety of configurations to suit most processes heat transfer requirements. The advantages of PHEs, and associated heat transfer enhancement techniques, extend far beyond energy efficiency. Lower capital cost, reduced plant size, and increased safety are typical of the benefits arising from the use of PHEs. Plate heat exchangers can replace some normal size heat exchangers bringing advantages and performance

c. Scraped surface heat exchangers

One type of heat exchanger, that finds considerable use in the food processing industry particularly for products of higher viscosity, consists of a jacketed cylinder with an internal cylinder concentric to the first and fitted with scraper blades. The blades rotate, causing the fluid to flow through the annular space between the cylinders with the outer heat transfer surface constantly scraped. Coefficients of heat transfer vary with speeds of rotation but they are of the order of 900-4000 J m^{-2} s^{-1} $°C^{-1}$. These machines are used in the freezing of ice cream and in the cooling of fats during margarine manufacture. Liquid foods that contain large solid particles are heated in **scraped-surface heat exchanger**s. These heat exchangers use blades to continuously scrape the inside surface of the heating chamber. The scraping action protects highly viscous foods from being burned on the heating surface.

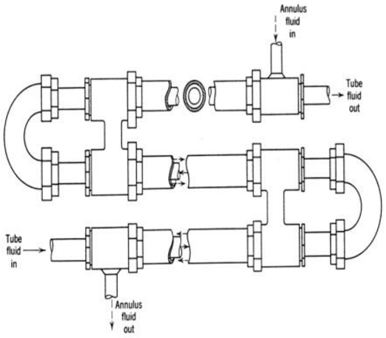


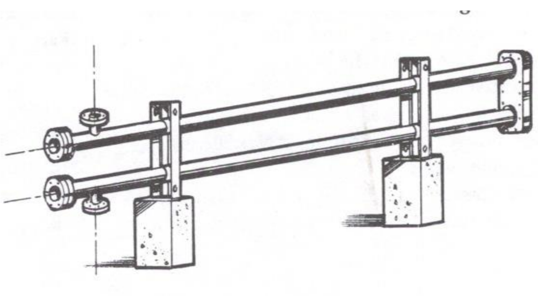
The **dynamic scraped surface heat exchanger** (DSSHE) was designed to face some problems found in other types of heat exchangers. They increase heat transfer by:

- removing the fouling layers,
- increasing turbulence in case of high viscosity flow,
- Avoiding the generation of ice and other process by-products.

The dynamic scraped surface heat exchangers incorporate an internal mechanism which periodically removes the product from the heat transfer wall. The product side is scraped by blades attached to a moving shaft or frame. The blades are made of a rigid plastic material to prevent damage to the scraped surface. This material is FDA approved in the case of food applications.

d. Double pipe heat exchangers





d. Double Pipe Exchanger

Two sets of concentric pipes, two connecting tees, and a return head and a return bend. A device whose purpose is the transfer of thermal energy between two fluids. Common applications: boilers, coolers, condensers, evaporators. Common design consists of tow fluids separated by a conducting medium.

Simplest type has one tube inside another - inner tube may have longitudinal fins on the outside However, most have a number of tubes in the outer tube - can have very many tubes thus becoming a shell-and-tube.

<u>Advantages of Double Pipe Heat Exchangers</u>

1. Double pipe heat exchanger consists of two concentric pipes are hot fluid, cold fluid.
2. Economically adaptable to service differentials. Ideal for wide temperature ranges and differentials.

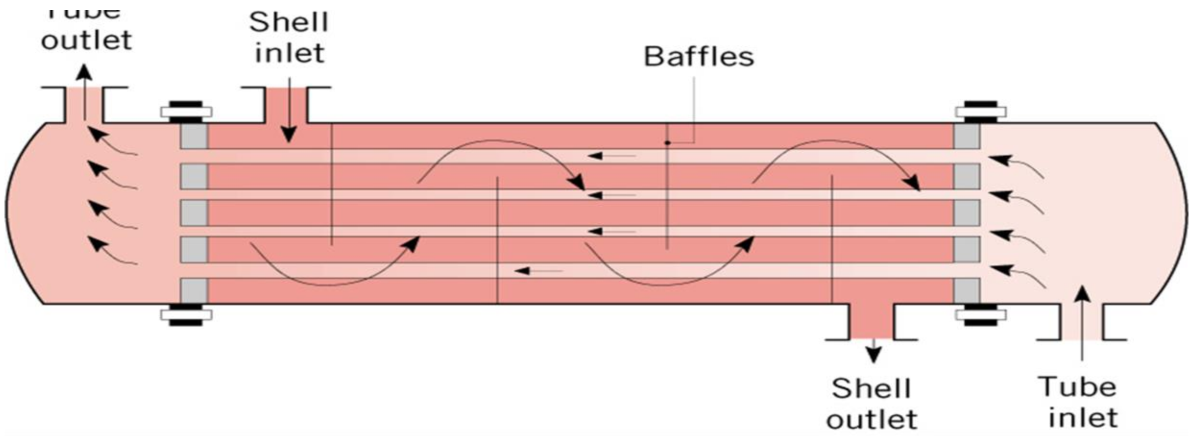
3. Provides shorter deliveries than shell and tube due to standardization of design and construction.
4. Operates in true counter current flow permitting extreme temperature cross.

e. Tubular heat exchangers

This type of heat exchanger are categorized in following types: Double Pipes heat Exchanger, Shell& Tube Heat Exchanger, Spiral Heat Exchanger, Cracked Surface Heat Exchanger.

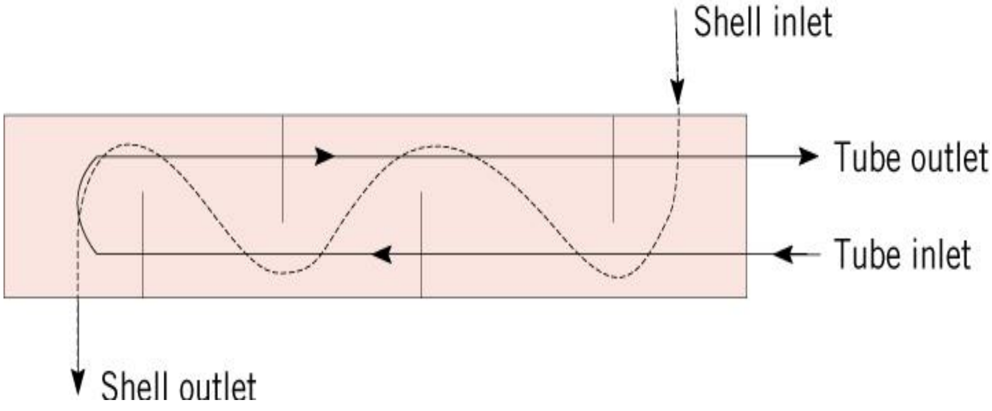
Shell and Tube Heat Exchangers

- The shell and tube heat exchanger is the most common style found in industry.
- As the tube-side flow enters the exchanger, flow is directed into tubes that run parallel to each other. these tubes run through a shell that has a fluid passing through it.
- Heat energy is transferred through the tube wall into the cooler fluid.
- Heat transfer occurs primarily through conduction and convection.



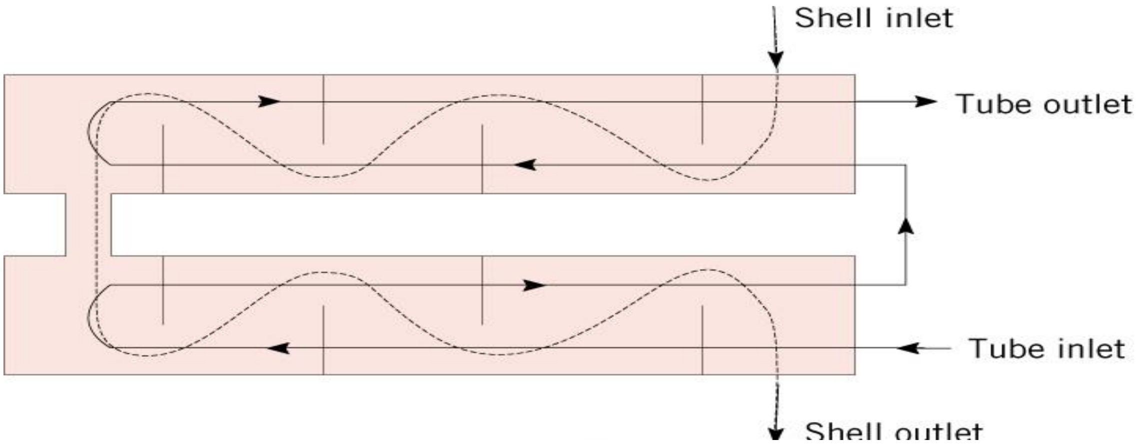
One Shell Pass and One Tube Pass

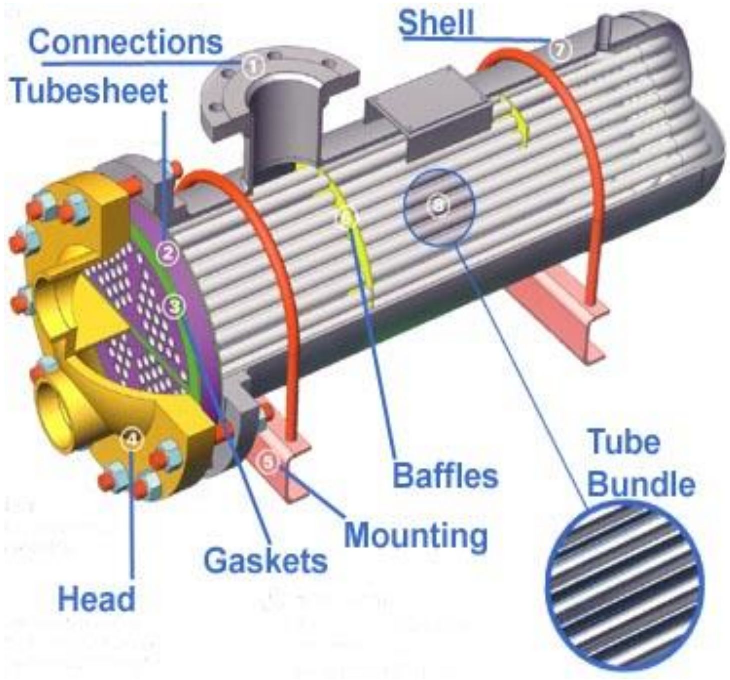
Baffles are used to establish a cross-flow and to induce turbulent mixing of the shell-side fluid.



One Shell Pass,Two Tube Passes

Two Shell Passes,Four Tube Passes





Main Parts:

1.Connections

2.Tube Sheets

3.Gaskets

4.Head

5.Mounting

6.Baffles

7. Shell

8.Tube bundle

Thermal design considerations

Thermal design of a shell and tube heat exchanger typically includes the determination of heat transfer area, number of tubes, tube length and diameter, tube layout, number of shell and tube passes, type of heat exchanger (fixed tube sheet, removable tube bundle etc), tube pitch, number of baffles, its type and size, shell and tube side pressure drop etc.

Shell

Shell is the container for the shell fluid and the tube bundle is placed inside the shell. Shell diameter should be selected in such a way to give a close fit of the tube bundle. The clearance between the tube bundle and inner shell wall depends on the type of exchanger (**[2]; page 647)** . The shells are usually fabricated from standard steel pipe with satisfactory corrosion allowance. The shell thickness of 3/8 inch for the shell ID of 12-24 inch can be satisfactorily used upto 300 psi of operating pressure.

Tube

Tubes OD of ¾ and 1" are very common to design a compact heat exchanger. The most efficient condition for heat transfer is to have the maximum number of tubes in the shell to increase turbulence. The tube thickness should be good enough to withstand the internal pressure along with the adequate corrosion allowance. The tube thickness is expressed in terms of BWG (Birmingham Wire Gauge) and true outside diameter (OD). The tube length of 6, 8, 12, 16, 20 and 24 ft are preferably used. Longer tube reduces shell diameter with the expense of higher shell pressure drop. Finned tubes are also used when fluid with low heat transfer coefficient flows in the shell side. Stainless steel, admiralty brass, copper, bronze and alloys of copper-nickel are the commonly used tube materials:

Tube pitch, tube-layout and tube-count

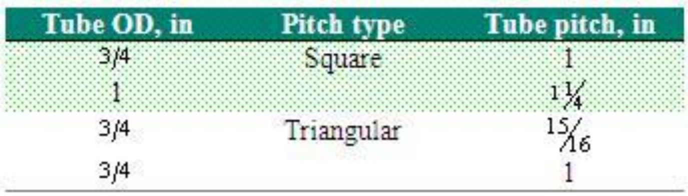
Tube pitch is the shortest centre to centre distance between the adjacent tubes. The tubes are generally placed in square or triangular patterns as shown in the **Figure 5** . The widely used tube layouts are illustrated in **Table 2.**

The number of tubes that can be accommodated in a given shell ID is called tube count. The tube count depends on the factors like shell ID, OD of tube, tube pitch, tube layout, number of tube passes, type of heat exchanger and design pressure.

Tube passes

The number of passes is chosen to get the required tube side fluid velocity to obtain greater heat transfer co-efficient and also to reduce scale formation. The tube passes vary from 1 to 16. The tube passes of 1, 2 and 4 are common in application. The partition built into exchanger head known as partition plate (also called pass partition) is used to direct the tube side flow.

- **Table 2** . Common tube layouts.



Tube OD, in	Pitch type	Tube pitch, in
3/4	Square	1
1		1¼
3/4	Triangular	15/16
3/4		1

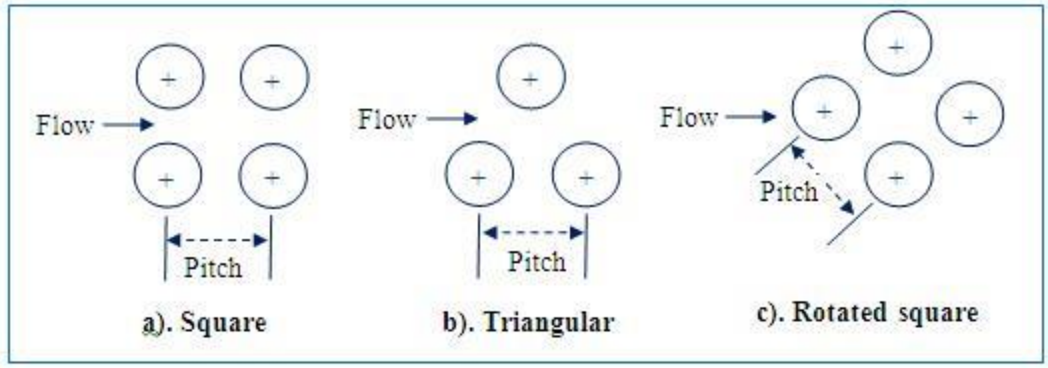


Figure 5 . Heat exchanger tube-layouts.

Tube sheet

The tubes are fixed with tube sheet to form the barrier between the tube and shell fluids. The tubes are generally fixed with the tube sheet using ferrule and a soft metal packing ring. The tube sheet thickness should be greater than the tube outside diameter to make a good seal. The recommended standards (IS:4503 or TEMA) should be followed to select the minimum tube sheet thickness.

Baffles

Baffles are used to increase the fluid velocity by diverting the flow across the tube bundle to obtain higher transfer co-efficient. The distance between adjacent baffles is called baffle-spacing. The baffle spacing of 0.2 to 1 times of the inside shell diameter is very common. The baffles are positioned by means of baffle spacers. Closer baffle spacing gives greater transfer co-efficient by inducing higher turbulence. The pressure drop is more with closer baffle spacing. The various types of baffles are schematized in **Figure 6** . In case of cut-segmental baffle, a segment (called baffle cut) is removed to form the baffle expressed as a percentage of the baffle diameter. Baffle cuts from 15 to 45% are normally used. A baffle cut of 20 to 25% provide a good heat-transfer within the reasonable pressure drop.

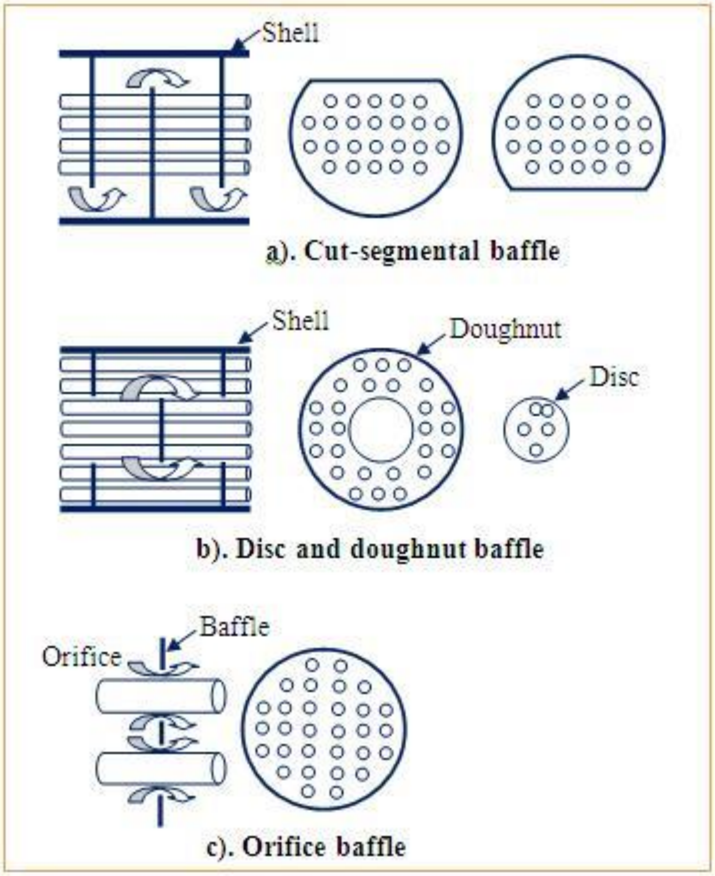


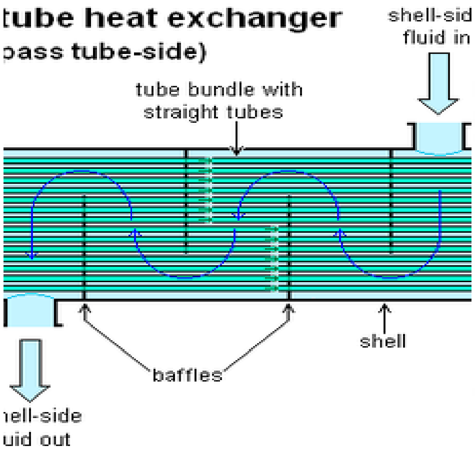
Figure 6. Different type of heat exchanger baffles: **a).** Cut-segmental baffle, **b).** Disc and doughnut baffle, **c).** Orifice baffle

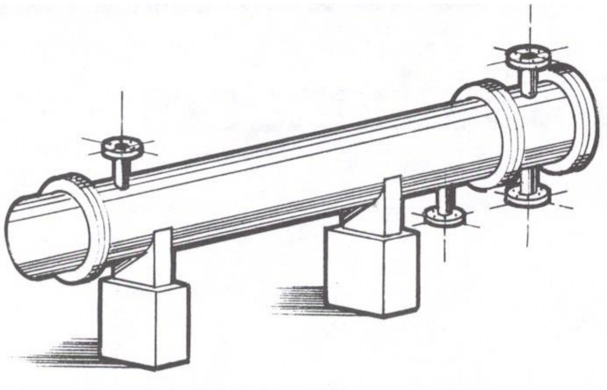
Fouling Considerations

The most of the process fluids in exchanger foul the heat transfer surface. The material deposited reduces the effective heat transfer rate due to relatively low thermal conductivity. Therefore, net heat transfer should the increased to compensate the reduction in performance during operation. Fouling of exchanger increases the cost of (i) construction due to oversizing, (ii) additional energy due to poor exchanger performance and (iii) cleaning to remove deposited materials. The duplicate exchanger may be required during cleaning for uninterrupted service.

Shell and tube heat exchanger

Shell and tube heat exchangers consist of a series of tubes. One set of these tubes contains the fluid that must be either heated or cooled. The second fluid runs over the tubes that are being heated or cooled so that it can either provide the heat or absorb the heat required. A set of tubes is called the tube bundle and can be made up of several types of tubes: plain, longitudinally finned. Shell and tube heat exchangers are typically used for high-pressure applications (with pressures greater than 30 bar and temperatures greater than 260 °C). This is because the shell and tube heat exchangersare robust due to their shape. **Shell and tube heat exchanger** units can be designed for almost all condition of temperature and pressure. In extreme cases, high pressure may impose a limitation by fabrication problems associate with material thickness





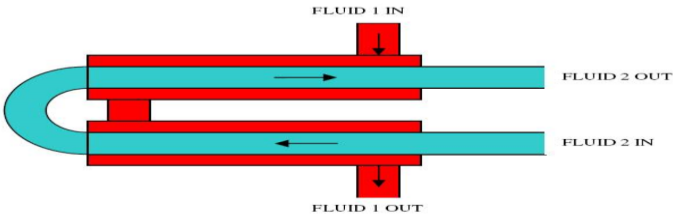
a. Shell and Tube Exchanger

Double pipe heat exchanger

They consist of one pipe concentrically located inside a second larger pipe. In this heat exchanger the fluid to be cooled or heated passes through the tube 2(green) and the other fluid is passed through tube 1 (red) to absorb or release the heat.

Advantages: Cheap for both design and maintenance.
Disadvantages: Low efficiency and requires large space.

Mainly used in pasteurization, digester heating, heat recovery, pre-heating, effluent cooling.



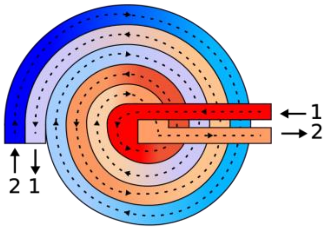
Spiral heat exchanger

A spiral heat exchanger (SHE), may refer to a helical (coiled) tube configuration efficient use of space. They can be easily cleaned.

A Spiral Heat Exchangers (or SHE) is a coiled tube arrangement, with two channels coiled one around the other. These two channels operate in a counter-flow arrangement, offering excellent turn down ratios, while optimizing flow patterns which in turn, enhance heat transfer. spiral heat exchangers are mainly used in pasteurization, recuperators (Exhaust and Air Handling Systems),sludge treatment (Thermal depolymerisation) Selection of spiral heat exchangers is based on cost, high/low pressure limits, thermal performance, temperature ranges, product mix (liquid/liquid, particulates or high-solids liquid),pressure drops across the exchanger, fluid flow capacity, clean ability, maintenance and repair, materials required for construction, ability and ease of future expansion Spiral heat exchangers can be used in most applications in the chemical process industry

In many difficult applications where fouling and plugging are problems, a standard shell and tube design may not be effective

While a spiral heat exchanger often has a higher initial cost, it may provide a lower life cycle cost due to lower fouling rates and ease of maintenance



<u>Contact type and Non-contact type</u>

<u>Contact type</u>

In Direct Contact type of heat Exchanger heat is directly transferred between Hot and Cold fluids.

There is no separating wall between the hot and cold fluids.

Most direct contact heat exchangers fall under the Gas- Liquid category, where heat is transferred between a gas and liquid in the form of drops, films or sprays.

Properties of both fluids, such as viscosity, thermal conductivity, density, specific heat and reaction on the metal in contact are essential to determine the pumping, thermal requirement and material construction.

In a direct-contact exchanger, two fluid streams come into direct contact, exchange heat, and are then separated.

Common applications of a direct-contact exchanger involve mass transfer in addition to heat transfer, such as in evaporative cooling and rectification.

However, the applications are limited to those cases where a direct contact of two fluid streams is permissible.

However direct contact are in three classes

a) Immiscible Fluid Exchangers
b) Gas–Liquid Exchangers
c) Liquid–Vapor Exchangers

Immiscible Fluid Exchangers

In this type, two immiscible fluid streams are brought into direct contact.

These fluids may be single-phase fluids, or they may involve condensation or vaporization.

Condensation of organic vapors and oil vapors with water or air are typical examples.

Gas–Liquid Exchangers

In this type, one fluid is a gas (more commonly, air) and the other a low-pressure liquid (more commonly, water) and are readily separable after the energy exchange.

In either cooling of liquid (water) or humidification of gas (air) applications, liquid partially evaporates and the vapor is carried away with the gas.

In these exchangers, more than 90% of the energy transfer is by virtue of mass transfer (due to the evaporation of the liquid), and convective heat transfer is a minor mechanism.

A ‘‘wet’’ (water) cooling tower with forced- or natural-draft airflow is the most common application.

Other applications are the air-conditioning spray chamber, spray drier, spray tower, and spray pond.

Liquid–Vapor Exchangers

In this type, typically steam is partially or fully condensed using cooling water, or water is heated with waste steam through direct contact in the exchanger.

Noncondensables and residual steam and hot water are the outlet streams.

Common examples are desuperheaters and open feed water heaters (also known as deaerators) in power plants.

Compared to indirect contact recuperators and regenerators, in direct-contact heat exchangers,

1. very high heat transfer rates are achievable,
2. the exchanger construction is relatively inexpensive, and
3. the fouling problem is generally nonexistent, due to the absence of a heat transfer surface (wall) between the two fluids.

Non-Contact type

- In an Indirect Contact Type Heat Exchanger, the fluid stream remains separated. There is separating wall between the hot and cold fluids.
- The heat transfer in this type of heat exchanger takes place continuously through a dividing wall.
- Most indirect contact heat exchangers fall under those gases and liquids that are soluble in nature.
- The fluid streams remain separate and the heat transfers continuously through a dividing wall into and out of the wall in a transient manner.

Non-contact are in three classes

a) Direct transfer type heat exchanger
b) Storage type heat exchanger
c) Fluidized bed heat exchanger

a) Direct Transfer Type Heat Exchanger

- In this, type heat transfers continuously from the hot fluid to the cold fluid through a dividing wall.
- There is no direct mixing of the fluids because each fluid flows in separate fluid passages.
- It is also known as recuperators. Examples, tubular exchangers, plate and frame heat exchangers and extended surface exchangers.

Tubular

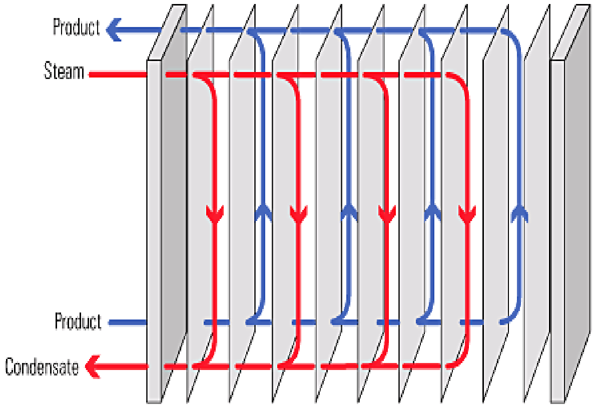
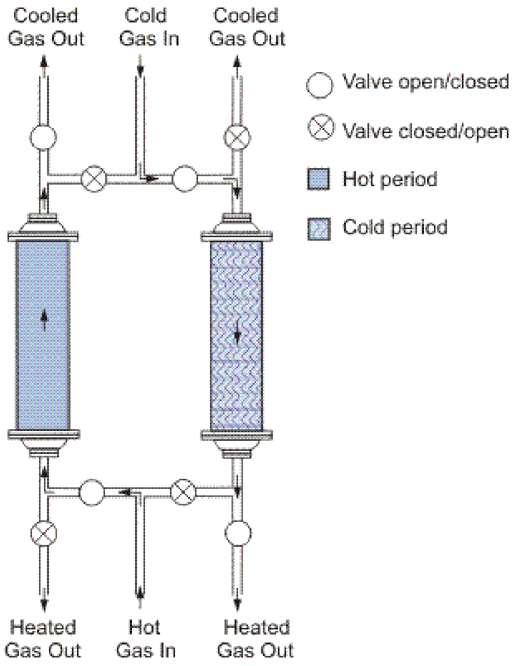


Plate and Frame

b) Storage Type Heat Exchanger (Regenerative Heat Exchanger)

- In a storage type exchanger, both fluids flow alternatively through the same flow passages, and hence heat transfer is intermittent.

- The heat transfer surface (or flow passages) is generally cellular in structure and is referred to as a matrix, or it is a permeable (porous) solid material, referred to as a packed bed.
- When hot gas flows over the heat transfer surface (through flow passages), the thermal energy from the hot gas is stored in the matrix wall, and thus the hot gas is being cooled during the matrix heating period.
- As cold gas flows through the same passages later (i.e., during the matrix cooling period), the matrix wall gives up thermal energy, which is absorbed by the cold fluid.
- Thus, heat is not transferred continuously through the wall as in a direct-transfer type exchanger (recuperator), but the corresponding thermal energy is alternately stored and released by the matrix wall.



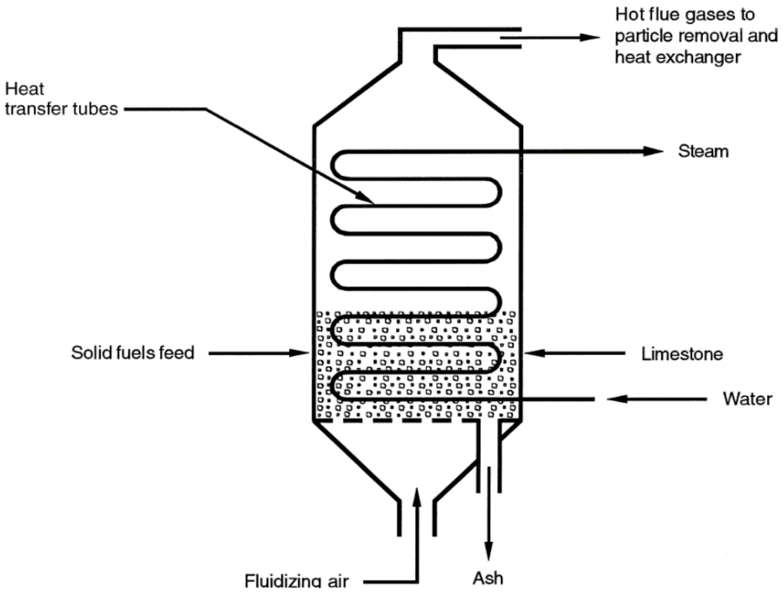
Fixed Bed Regenerator

b) **Storage Type Heat Exchanger (Regenerative Heat Exchanger)**

  a. Regenerative heating was one of the most important technologies developed during the Industrial Revolution when it was used in the hot blast process on blast furnaces.
  b. It was later used in glass and steel making, to increase the efficiency of open hearth furnaces, and in high pressure boilers and chemical and other applications, where it continues to be important today.

c. **Fluidized bed heat exchanger**

In a fluidized-bed heat exchanger, one side of a two-fluid exchanger is immersed in a bed of finely divided solid material, such as a tube bundle immersed in a bed of sand or coal particles. The common applications of the fluidized-bed heat exchanger are drying, mixing, adsorption, reactor engineering, coal combustion, and waste heat recovery

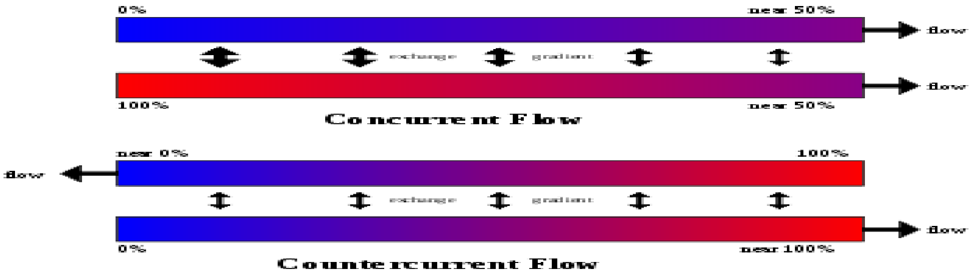


Difference between Direct and Indirect Heat Exchanger

Direct Heat Exchanger	**Indirect Heat Exchanger**
Not have a separating wall between two fluids.	Have a separating wall between two fluids.
Mostly used for those gases and liquids that are not soluble in nature	Used for those gases and liquids that are soluble in nature.

Mostly use drops and steam for heat transfer	There is no use of steam and drops

Co-current and counter-current flow in heat exchangers



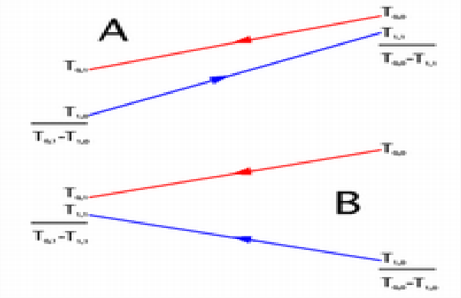
Co-current and countercurrent exchange mechanisms
the magnitude of the property to be exchanged, is represented by shading. The direction of transfer across the barrier is from the greater to the lesser magnitude.

In the co-current flow exchange mechanism, the two fluids flow in the same direction.

As the Co-current and countercurrent exchange mechanisms diagram showed, a co-current exchange system has a variable gradient over the length of the exchanger. With equal flows in the two tubes, this method of exchange is only capable of moving half of the property from one flow to the other, no matter how long the exchanger is.

If each stream changes its property to be 50% closer to that of the opposite stream's inlet condition, exchange will stop when the point of equilibrium is reached, and the gradient has declined to zero. In the case of unequal flows, the equilibrium condition will occur somewhat closer to the conditions of the stream with the higher flow.

Co-current flow examples



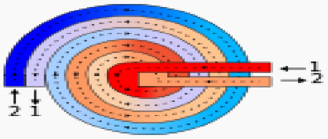
Co-current and Countercurrent heat exchange

A **co-current heat exchanger** is an example of a co-current flow exchange mechanism. Two tubes have a liquid flowing in the same direction. One starts off hot at 60 °C, the second cold at 20 °C. A thermoconductive membrane or an open section allows heat transfer between the two flows.

The hot fluid heats the cold one, and the cold fluid cools down the warm one. The result is thermal equilibrium: Both fluids end up at around the same temperature: 40 °C, almost exactly between the two original temperatures (20 and 60 °C). At the input end, there is a large temperature difference of 40 °C and much heat transfer; at the output end, there is a very small temperature difference (both are at the same temperature of 40 °C or close to it), and very little heat transfer if any at all. If the equilibrium—where both tubes are at the same temperature—is reached before the exit of the liquid from the tubes, no further heat transfer will be achieved along the remaining length of the tubes.

A similar example is the **co-current concentration exchange**. The system consists of two tubes, one with brine (concentrated saltwater), the other with freshwater (which has a low concentration of salt in it), and a semi permeable membrane which allows only water to pass between the two, in an osmotic process. Many of the water molecules pass from the freshwater flow in order to dilute the brine, while the concentration of salt in the freshwater constantly grows (since the salt is not leaving this flow, while water is). This will continue, until both flows reach a similar dilution, with a concentration somewhere close to midway between the two original dilutions. Once that happens, there will be no more flow between the two tubes, since both are at a similar dilution and there is no more osmotic pressure.

Countercurrent flow



Spiral counter-current heat exchange schematic

In countercurrent flow, the two flows move in opposite directions.

Two tubes have a liquid flowing in opposite directions, transferring a property from one tube to the other. For example, this could be transferring heat from a hot flow of liquid to a cold one, or transferring the concentration of a dissolved solute from a high concentration flow of liquid to a low concentration flow.

The counter-current exchange system can maintain a nearly constant gradient between the two flows over their entire length of contact. With a sufficiently long length and a sufficiently low flow rate this can result in almost all of the property transferred. So, for example, in the case of heat exchange, the exiting liquid will be almost as hot as the original incoming liquid's heat.

Countercurrent flow examples

In a **countercurrent heat exchanger**, the hot fluid becomes cold, and the cold fluid becomes hot.

In this example, hot water at 60 °C enters the top pipe. It warms water in the bottom pipe which has been warmed up along the way, to almost 60 °C. A minute but existing heat difference still exists, and a small amount of heat is transferred, so that the water leaving the bottom pipe is at close to 60 °C. Because the hot input is at its maximum temperature of 60 °C, and the exiting water at the bottom pipe is nearly at that temperature but not quite, the water in the top pipe can warm the one in the bottom pipe to nearly its own temperature. At the cold end—the water exit from the top pipe, because the cold water entering the bottom pipe is still cold at 20 °C, it can extract the last of the heat from the now-cooled hot water in the top pipe, bringing its temperature down nearly to the level of the cold input fluid (21 °C).

The result is that the top pipe which received hot water, now has cold water leaving it at 20 °C, while the bottom pipe which received cold water, is now emitting hot water at close to 60 °C. In effect, most of the heat was transferred.

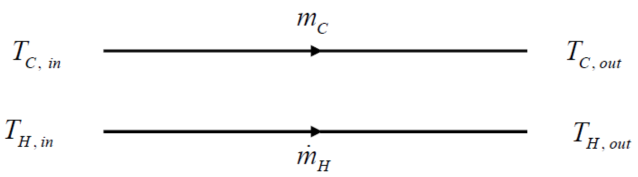
Conditions for higher transfer results

It should be noted that nearly complete transfer in systems implementing countercurrent exchange, is only possible if the two flows are, in some sense, "equal".

For a maximum transfer of substance concentration, an equal flowrate of solvents and solutions is required. For maximum heat transfer, the average specific heat capacity and the mass flow rate must be the same for each stream. If the two flows are not equal, for example if heat is being transferred from water to air or vice versa, then, similar to co-current exchange systems, a variation in the gradient is expected because of a buildup of the property not being transferred properly.

Types of flows in heat exchangers

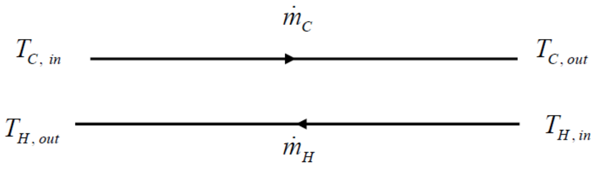
Co-current flow



where m_c – mass flow rate in cold stream

m_h – mass flow in hot stream

Countercurrent flow



Classification of Heat Exchangers

Heat exchangers are generally classified according to the relative directions of hot and cold fluids:

(a) Parallel Flow – the hot and cold fluids flow in the same direction. Fig 3.2 depicts such a heat exchanger where one fluid (say hot) flows through the pipe and the other fluid (cold) flows through the annulus.

(b) Counter Flow – the two fluids flow through the pipe but in opposite directions. A common type of such a heat exchanger is shown in Fig. 3.3. By comparing the temperature distribution of the two types of heat exchanger we find that the temperature difference between the two fluids is more uniform in counter flow than in the parallel flow. Counter flow exchangers give the maximum heat transfer rate and are the most favoured devices for heating or cooling of fluids.

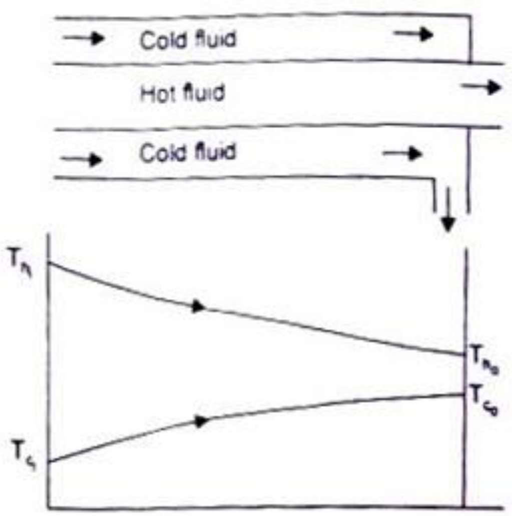


Fig 3.2 Parallel flow heat exchanger with temperature distribution

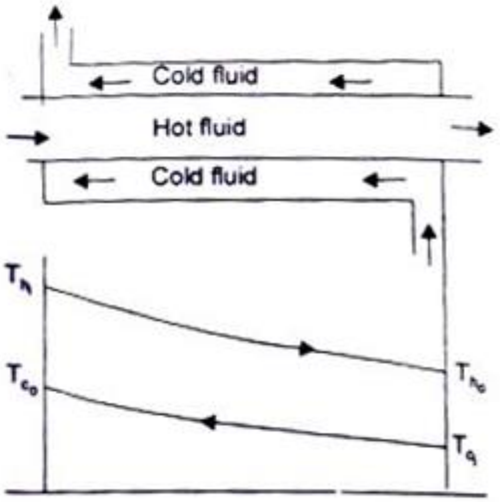


Fig 3.3 Counter-flow heat exchanger with temperature distribution

When the two fluids flow through the heat exchanger only once, it is called one-shell-pass and one-tube-pass as shown in Fig. 3.2 and 3.3. If the fluid flowing through the tube makes one pass through half of the tube, reverses its direction of flow, and makes a second pass through the remaining half of the tube, it is called 'one-shell-pass, two-tube-pass' heat exchanger,fig 3.4. Many other possible flow arrangements exist and are being used. Fig. 10.5 depicts a 'two-shell-pass, four-tube-pass' exchanger.

(c) Cross-flow - A cross-flow heat exchanger has the two fluid streams flowing at right angles to each other. Fig. 3.6 illustrates such an arrangement An automobile radiator is a good example of cross-flow exchanger. These exchangers are 'mixed' or 'unmixed' depending upon the mixing or not mixing of either fluid in the direction transverse to the direction of the flow stream and the analysis of this type of heat exchanger is extremely complex because of the variation in the temperature of the fluid in and normal to the direction of flow.

(d) Condenser and Evaporator - In a condenser, the condensing fluid temperature remains almost constant throughout the exchanger and temperature of the colder fluid gradually increases from the inlet to the exit, Fig. 3.7 (a). In an evaporator, the temperature of the hot fluid gradually decreases from the inlet to the outlet whereas the temperature of the colder fluid remains the same during the evaporation process, Fig. 3.7(b). Since the temperature of one of the fluids can be treated as constant, it is immaterial whether the exchanger is parallel flow or counter flow.

(e) Compact Heat Exchangers - these devices have close arrays of finned tubes or plates and are typically used when atleast one of the fluids is a gas. The tubes are either flat or circular as shown in Fig. 10.8 and the fins may be flat or circular. Such heat exchangers are used to a chieve a very large (≥ 700 m^2/mJ) heat transfer surface area per unit volume. Flow passages are typically small and the flow is usually laminar.

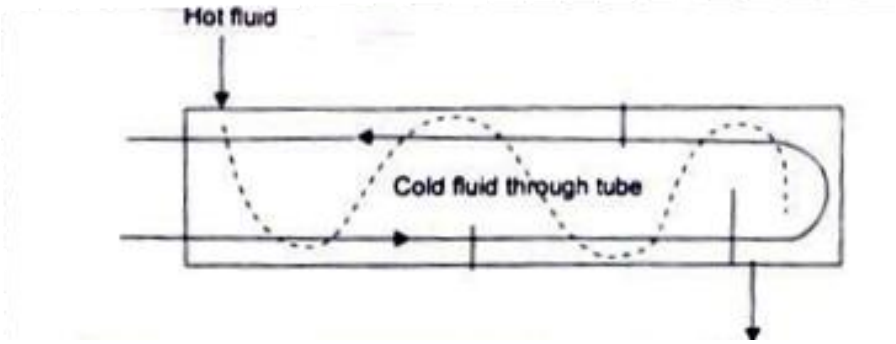


Fig 3.4: multi pass exchanger one shell pass, two shell pass

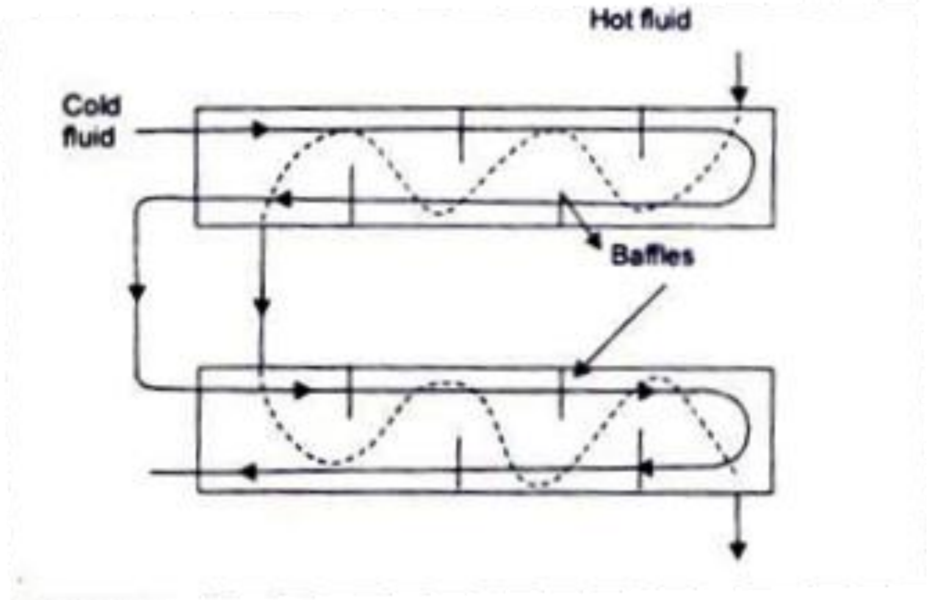


Fig 3.5: Two shell passes, four-tube passes heat exchanger (baffles increases the convection coefficient of the shell side fluid by inducing turbulance and a cross flow velocity component)

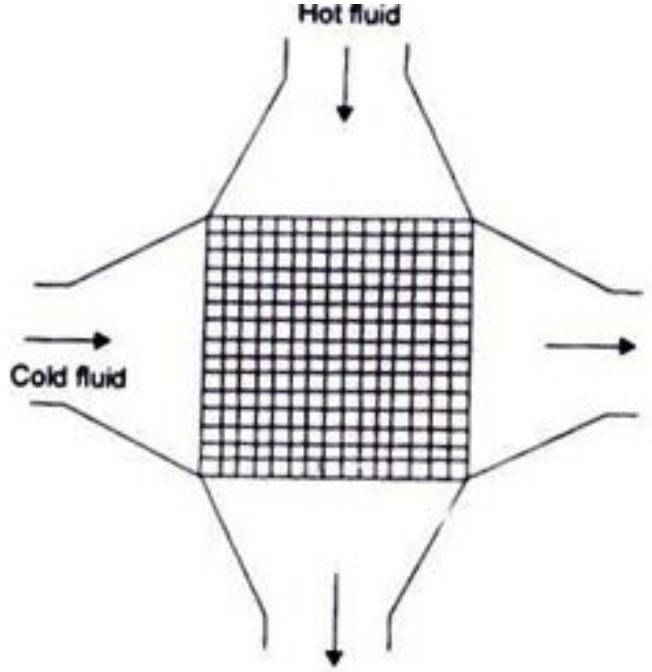


Fig 3.6: A cross-flow exchanger

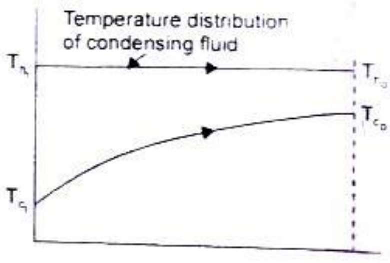


Fig. 10.7 (a) A condenser

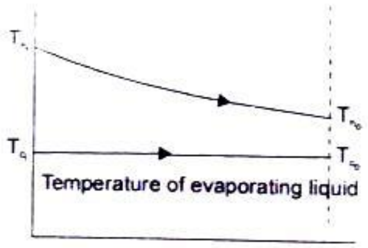


Fig. 10.7 (b) An evaporator

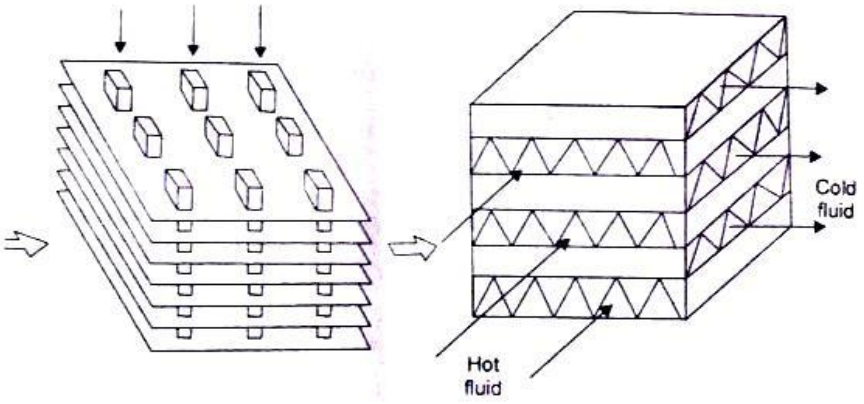


Fig. 3.8 Compact heat exchangers: (a) flat tubes, continuous plate fins, (b) plate fin (single pass)

Condenser

The change from liquid phase to vapor phase is called vaporization and the reverse phase transfer is condensation. The change from liquid to vapor or vapor to liquid occurs at one temperature (called saturation or equilibrium temperature) for a pure fluid compound at a given pressure. The industrial practice of vaporization and condensation occurs at almost constant pressure; therefore the phase change occurs isothermally.

Condensation occurs by two different physical mechanisms i.e. **drop-wise condensation** and **film condensation** .

The nature of the condensation depends upon whether the condensate (liquid forms from vapor) wets or does not wet the solid surface. If the condensate wets the surface and flows on the surface in the form of a film, the process is called film condensation. When the condensate does not wet the solid surface and the condensate is accumulated in the form of droplets, the mechanism is drop-wise condensation. **Heat transfer coefficient is about 4 to 8 times higher for drop wise condensation.** The condensate forms liquid film on the bare-surface in case of film condensation. The heat transfer coefficient is lower for film condensation due to the liquid film resistance.

Dropwise condensation occurs usually on new, clean and polished surfaces. The equipment used for condensation heat transfer is called condenser. In industrial condensers, film condensation normally occurs.

Types of condensers

There two general types of condensers:

- **i.** Vertical condenser

  Downflow vertical condenser: The vapor enters at the top of condenser and flows down inside tubes. The condensate drains from the tubes by gravity and vapor induced shear **(Figure 7)** .

  Upflow vertical condenser: In case of upflow condenser, the vapor enters at the bottom and flows upwards inside the tubes. The condensate drains down the tubes by gravity only.

  ii. Horizontal condenser: The condensation may occur inside or outside the horizontal tubes (**Figure 8**). Condensation in the tube-side is common in air-cooled condensers. The main disadvantage of this type of condenser is that the liquid tends to build up in the tubes. Therefore the effective heat transfer co-efficient is reduced significantly.

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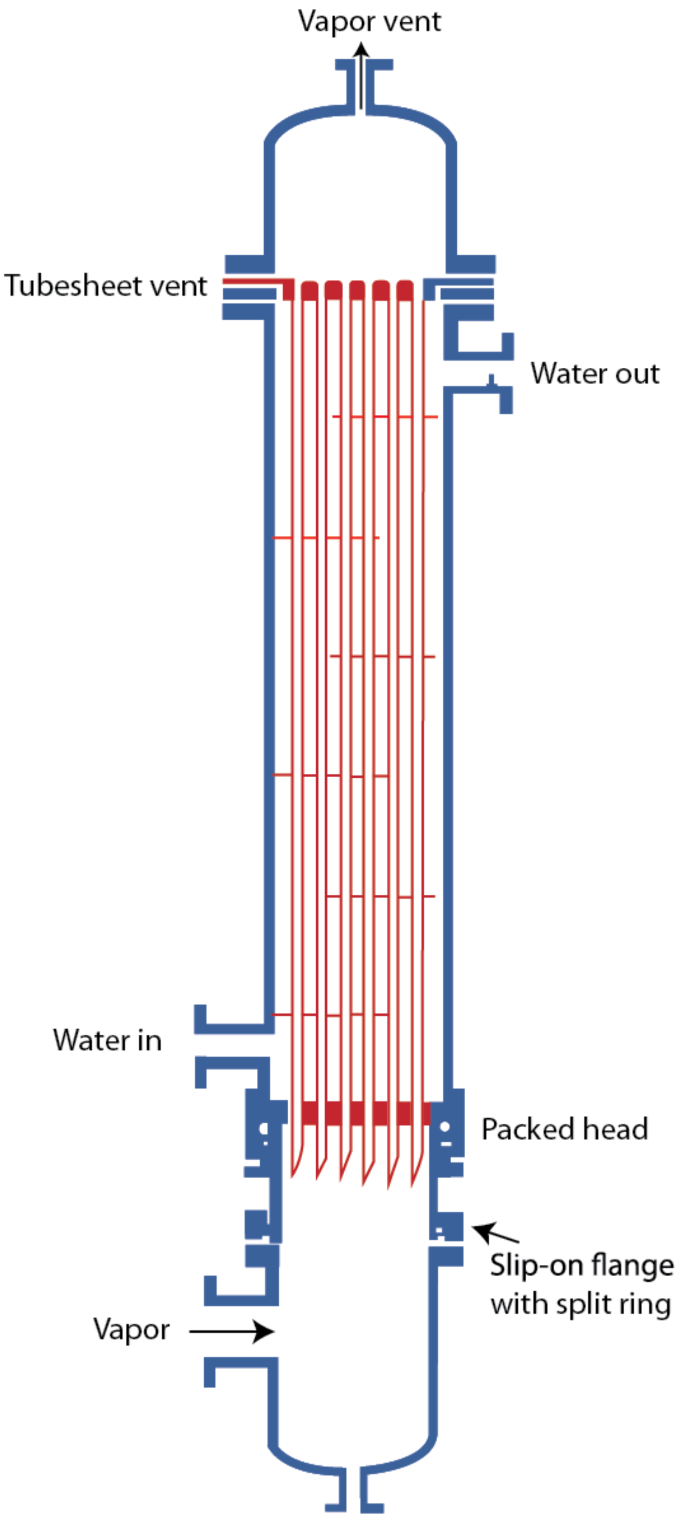


Figure 7 . Downflow vertical condenser with condensation inside tube **[5]** .

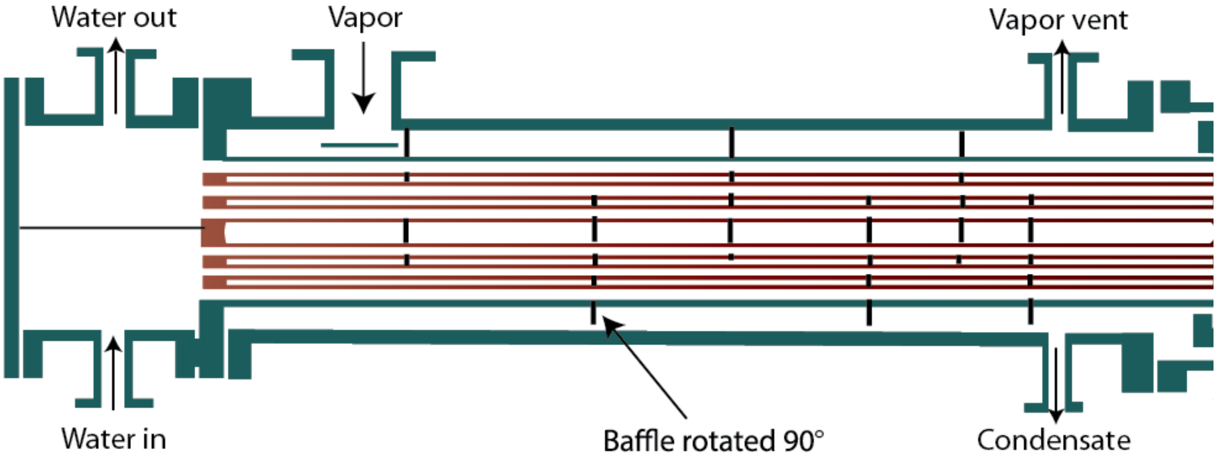


Figure 8. Horizontal condenser with condensation outside horizontal tubes **[5]** .

Condenser design considerations

The design of condenser is similar to a typical shell and tube exchangers. A condenser must have a vent for removal of non-condensable gas. The non-condensable gas decreases the heat transfer rate by reducing condensation temperature. Condenser usually uses a wider baffle spacing of B = D_s (ID of shell). Vertical cut-segmental baffles are generally used in condensers for side to side vapor flow not for top to bottom. An opening at the bottom of the baffles is provided to allow draining of condensate.

Mean temperature difference

The condensation occurs almost at a fixed temperature (isothermally) at constant pressure for a pure saturated vapor compound. The logarithmic mean temperature difference can be used for condenser design. **No correction factor for multiple pass condensers is required** . The logarithmic mean temperature difference:

$$LMTD = \frac{(T_{sat} - t_1) - (T_{sat} - t_2)}{\ln \dfrac{(T_{sat} - t_1)}{(T_{sat} - t_2)}} = \frac{(t_2 - t_1)}{\ln \dfrac{(T_{sat} - t_1)}{(T_{sat} - t_2)}}$$

Where, T_{sat} = *Saturation vapor temperature*

t_1 = *Coolant inlet temperature*

t_2 = *C oolant outlet temperature*

Reboilers

Classification of reboilers

There are three major types of reboilers:

i. Thermosyphon natural circulation reboiler: The boiling occurs inside the tubes in vertical thermosyphon reboiler and inside shell in horizontal thermosyphon reboiler (**Figure 10)** . In vertical thermosyphon reboiler, the liquid circulation occurs due to density difference between vapor-liquid mixture (two phase) in the exchanger from the reboiler and the liquid through the downcomer to the reboiler.

Advantages: most economical because no pump is required.

Limitations: not suitable for heavily viscous fluid; high construction cost for the installation of the column base at suitable elevation to get thermosyphon effect; not suitable for low temperature difference processes due to boiling point elevation imposed by static head.

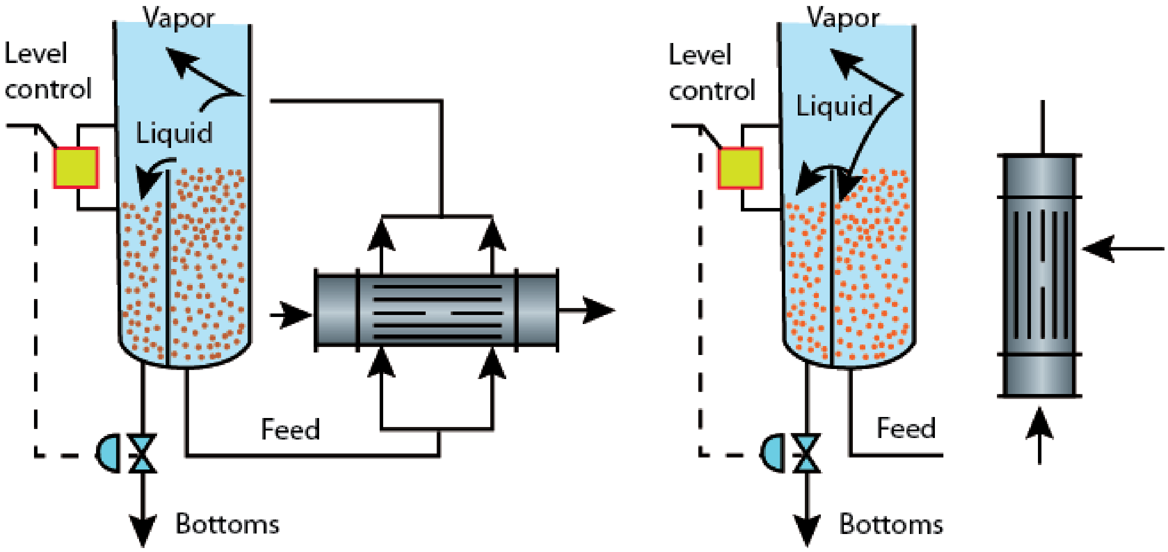


Figure 10. Thermosyphon reboiler **[5]** .

ii. Forced circulation reboiler : The liquid is fed by means of a pump. Forced circulation reboilers with inside vertical or horizontal tubes boiling may be designed. Forced circulation reboilers are similar to vertical thermosiphon reboilers, except the pump is used for the recirculation of the liquid. To calculate the heat transfer coefficient it is generally assumed that, heat is transferred only by forced convection. The usual method of shell and tube exchanger design can be used.

Advantage: suitable for viscous and highly fouling fluids.

Disadvantage: high pumping and maintenance cost; pump is required to circulate the boiling liquid through the tubes and back into the column.

iii. Kettle reboiler : The tube bundle is immerged in a pool of liquid at the base in an oversize shell (**Figure 11**). Kettle reboiler is also called a "submerged bundle reboiler". The height of the tube bundle is usually 40-60% of the shell ID. The submergence of the tube bundle is assured by an overflow weir at height of typically 5-15 cm from the upper surface of topmost tubes.

Advantage: suitable for vacuum operation and high vaporization rate upto about 80% of the feed.

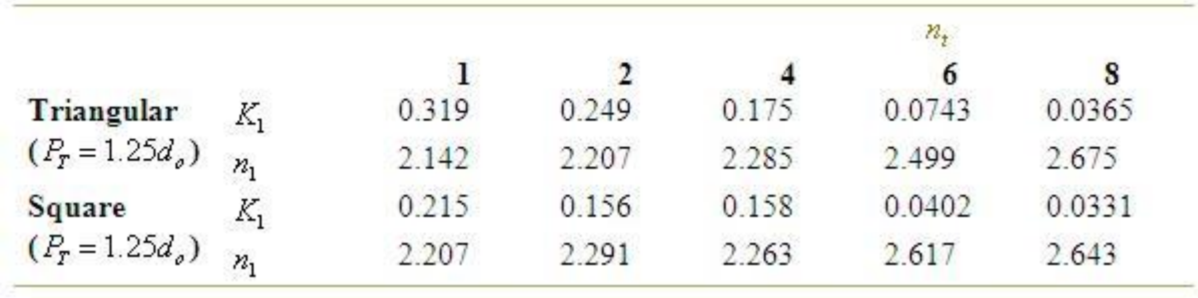
Limitations: low heat transfer rate than other types as there is no liquid circulation (low velocity); not appropriate for fouling fluids; kettle reboiler is not suitable for heat sensitive materials as higher residence time is required.

The bundle diameter D_b , can be obtained from the empirical equation **([2] page 647-649)** :

$$D_b = d_o \left(\frac{n_t}{K_1} \right)^{1/n}$$

where, D_b = bundle diameter [mm], n_t = number of tubes, d_o = tube outside diameter [mm]. The values of the constants k_1 and n_1 are in **Table 5.**

Table 5. Constants used to calculate the tube bundle diameter.



		n_t				
		1	**2**	**4**	**6**	**8**
Triangular	K_1	0.319	0.249	0.175	0.0743	0.0365
$(P_T = 1.25d_o)$	n_1	2.142	2.207	2.285	2.499	2.675
Square	K_1	0.215	0.156	0.158	0.0402	0.0331
$(P_T = 1.25d_o)$	n_1	2.207	2.291	2.263	2.617	2.643

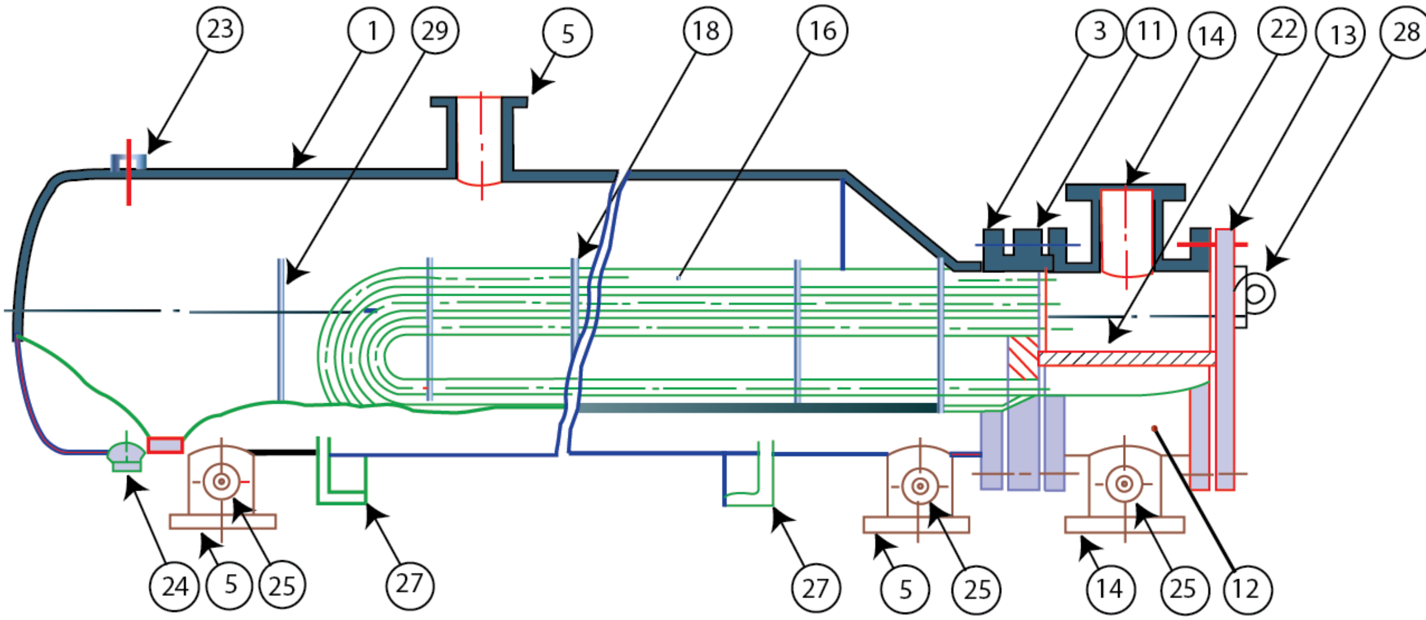


Figure 11 . Kettle type reboiler **[1]** .

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