

Heat Transfer

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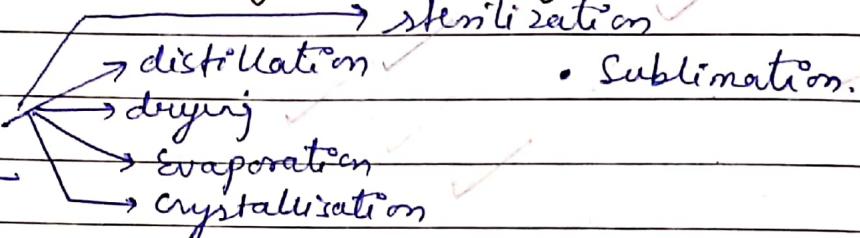
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- Heat is a form of energy. It is the transfer of heat. In any operation heat either enters the system or leaves the system.

The main objectives of heat transfer process are -

- 1) To Study different mode of heat transfer
- 2) To determine the rate of heating & cooling.
- 3) To plan changes in new heat transfer equipments.
- 4) To determine the efficiency of existing heat exchange equipments.

Applications



- ① Heat is required for drying of wet mass during production of tablet.
- ② During distillation, heat is required to convert liquid into vapour.
- ③ for diff processes such as boiling, fusion, etc, heat is also required.
- ④ for crystallization of drugs, supersaturation is achieved by heating saturated solution.
- ⑤ In case of steam distillation steam is reg. which is in direct contact with material.
- ⑥ Dry heat is required for sterilisation.
- ⑦ To prepare vegetable extract.

Mechanism of Heat Transfer

When we transfer heat to a material it flows from one region of high temp. to low temp. region.

There are 3 ways of heat transfer

• Radiation.

Conduction

- Takes place in solids or b/w 2 substances which are in physical contact.
- Mixing does not occur here.
- The better the conductor, sooner heat will be transferred.
- As we heat, particles get heated & acquire more energy & starts vibrating & they bump to neighbour particle & transfer some energy to them & to next & so on.
- It then continues & transmits energy from the hot end to the most coldest end of substance.
- Occurs in fluids of high viscosity.

Convection

- Energy transferred from hot places to cold places by mixing of warmer portion with cooler part of same material.
- ex - ① Water boiling in pan
 ② Water cycle process [natural convection].
- * If motion of fluid is due to mechanical means, then it is c/d forced convection.

Radiation

- It does not require any contact b/w heat source & heated object. as in case of previous ones.
- Heat transmitted by EM radiations.
- ex - ① Heat of sun
 ② Heat generated by filament of bulb.

Conduction

- When energy or heat is transferred, there occurs thermal conductivity.
- When thermal conductivity is higher, resistance will be less & more will be heat transfer.
- Heat flow occurs when there is a temp. gradient.
- More the temp. grad., more will be rate of heat transfr; until eqbm is reached.

$$\boxed{\text{Rate of Heat transfer} = \frac{\text{Driving force}}{\text{Resistance}} = \frac{\Delta t \text{ (temp. diff.)}}{R}}$$

Fourier's Law

- This law is used to calculate resistance.
- This law states that the rate of heat flow through a uniform material is proportional to the area (m^2) & temp. diff. while inv. prop. to the length of path of flow (m).

$$q \propto \frac{A \Delta t}{L}$$

$$\boxed{q = \frac{km A \Delta t}{L}} \quad -\textcircled{1}$$

Resistance = Thickness of surface

mean proportionality \times area of
constant surface

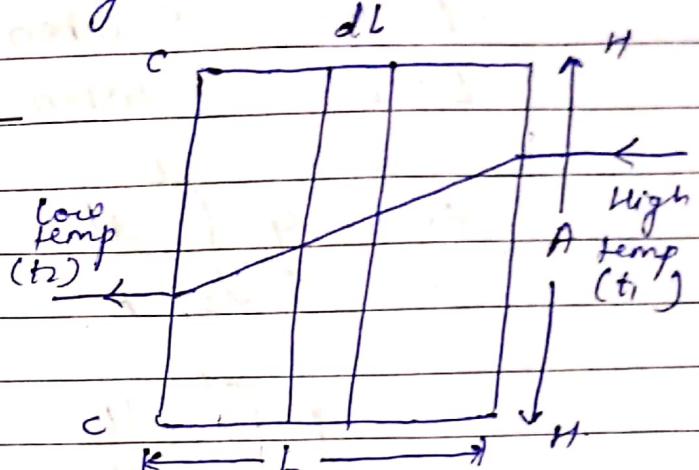
$$R = \frac{L}{km A}$$

Conduction of Heat Through a Metal wall

Rate of heat flow \propto Area \times temp diff
Thickness :

$$q \propto \frac{A \Delta t}{L}$$

$$q = \frac{k m A \Delta t}{L} \quad -\textcircled{1}$$



Consider a metal wall, whose

$$\text{Area} = A$$

$$\text{Thickness} = L$$

HH = Higher temp of uniform temp (t_1)

CC = lower of uniform temp (t_2)

$$\frac{dq}{dx} = -k m A \Delta t / L \quad -\textcircled{2}$$

-ve sign shows temp. gradient

In steady state -

$$\frac{dq}{dx} = q = \text{constant} = -k \frac{A \Delta t}{L} \quad -\textcircled{3}$$

Rearranging eq - \textcircled{3}

$$q \cdot \frac{dx}{A} = -k dt$$

Integrating the rearranged eq -

On integration

$$L = a \quad \text{when } t = t_1 \quad 4$$

$$L = L' \quad \text{when } t = t_2$$

$$\therefore q \int_0^L \frac{dL}{A} = - \int_{t_1}^{t_2} k \cdot dt$$

$$\therefore q \int_0^L \frac{dL}{A} = \int_{t_2}^{t_1} k \cdot dt$$

$$q \cdot \frac{L}{A} = km(t_1 - t_2)$$

$$\frac{qL}{A} = km(\Delta t) \quad \text{--- (5)}$$

Rearranging eq. (5)

$$q = \frac{kmA}{L} (\Delta t) \rightarrow \text{driving force}$$

Rearranging the above equation —

$$q = \frac{\Delta t}{L / kmA}$$

$$R = \frac{L}{kmA}$$

This law is only applicable in conduction where one atom transfers the heat to other atom.

#

Application of Fourier's Law

Thermal conductivity α

Thermal Resistance

Material

Copper

379

Silver

57

Steel

43

Aluminium

24.2

Stainless steel

17

Water

0.62

→ Liquids

Air

0.03

& solids

offer more resistance
than solids.

- The amount of heat transferred through a unit surface in unit time when Δt is constant.
- To conduct heat in a matter the outside of the material should be covered with metals such as stainless steel that offers more resistance of eliminate heat loss.

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Conduction Through a Pipe

Temp. inside = t_1 , K

Temp. outside = t_2 , K

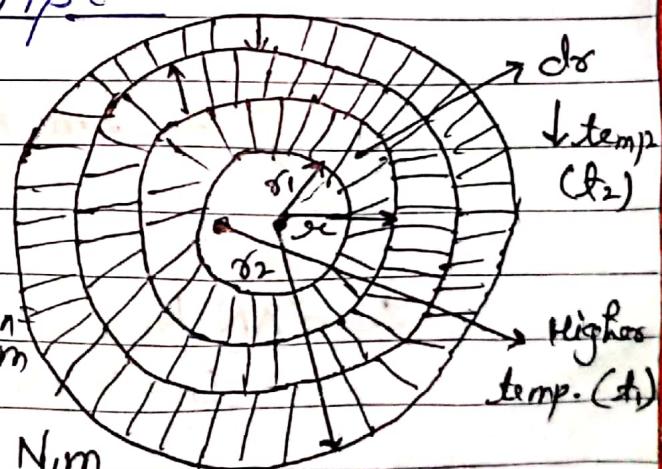
Radius = r_1 , m

Radius of the inner wall,

Thickness = d , m

Length of hollow cylinder = N , m

Thermal conductivity =



Acc. to Fourier's law,

$$q = -k_m \cdot 2\pi r N (t_1 - t_2)$$

dr

$$\Rightarrow \frac{dr}{A} = - \frac{k_m \cdot 2\pi N (t_1 - t_2)}{q} dt$$

When, $r = r_1$ & $t = t_1$,
 $\& r = r_2$ & $t = t_2$

$$\int_{r_1}^{r_2} \frac{dr}{r} = - \frac{k_m 2\pi N}{q} \int_{t_1}^{t_2} dt$$
$$= \frac{2\pi N}{q} \int_{t_2}^{t_1} k dt$$

$$\ln r_2 - \ln r_1 = \frac{2\pi N k m (t_1 - t_2)}{q}$$

$$q = \frac{2\pi N k m (t_1 - t_2)}{\ln(r_2/r_1)}$$

$$q = \frac{k \times \text{Area} \times \text{temp diff}}{\text{length}}$$

$$q = \frac{k m A m (t_1 - t_2)}{L}$$

$$\therefore \frac{2\pi N k m (t_1 - t_2)}{\ln(r_2/r_1)} = \frac{k m A m (t_1 - t_2)}{L}$$

$$\therefore A_m = \frac{2\pi N k_m (t_1 - t_2) L}{k_m (t_1 - t_2) \ln(\vartheta_2/\vartheta_1)}$$

$$A_m = \frac{2\pi N L}{\ln(\vartheta_2/\vartheta_1)} \quad (\because L = d\vartheta = \vartheta_2 - \vartheta_1)$$

$$A_m = \frac{2\pi N (\vartheta_2 - \vartheta_1)}{\ln(\vartheta_2/\vartheta_1)}$$

also, $A_m = 2\pi g_m N$

$$\therefore 2\pi g_m N = \frac{2\pi N (\vartheta_2 - \vartheta_1)}{\ln(\vartheta_2/\vartheta_1)}$$

$$\therefore g_m = \frac{(\vartheta_2 - \vartheta_1)}{\ln(\vartheta_2/\vartheta_1)}$$

$$\text{or } g_m = \frac{(\vartheta_2 - \vartheta_1)}{2.303 (\vartheta_2/\vartheta_1)}$$



Convection

- A process in which heat flow is achieved by actual mixing of warmer portions with cooler portion of same material.
- Actual mixing of its layer occurs.

Forced convection

Mixing of fluid attained with help of a stirrer or agitator or pumping the fluid for recirculation.
eg → forced circulation evaporators.

Natural Convection

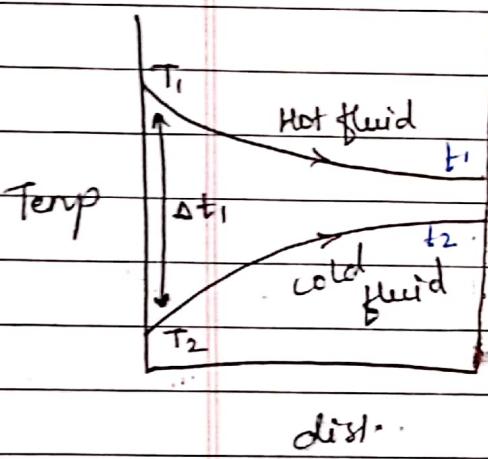
Mixing of fluid accomplished by the currents set up, when body of fluid is heated.
ex → pan evaporator.

Rate of Heat Transfer in Convection

$$(q = \text{overall heat transfer coefficient} \cdot A \Delta t)$$

↑ area of heat transfer surface

Parallel heat flow - variation in temperature



→ Heat transfer across metal surface from T to t

↓ Δt_2 → when hot & cold fluid enters from the same side the flow is called parallel flow.

→ Temp of hot fluid decreases & cold fluid increases.

T_1 = Ini. Temp of hot fluid before mixing

T_2 = Final Temp of hot fluid after mixing

t_1 = Ini. Temp. of cold fluid before mixn

t_2 = final. " " " " after "

Δt_1 = Initial Temp. dffnce

Δt_2 = Final Temp. dffnce

$$dq = UA \Delta t_m \quad \text{--- (1)}$$

On integration -

$$q = U A l \left[\frac{\Delta t_1 - \Delta t_2}{\ln \left[\frac{\Delta t_1}{\Delta t_2} \right]} \right] \quad \text{--- (2)}$$

L = length of pipe

a = Area of pipe ($al = A$)

on comparing eqn ① & ②

$$\Delta t'_{\text{atm}} = \frac{\nu g L}{A} \Delta t_1 - \Delta t_2$$

$$\ln \left[\frac{\Delta t_1}{\Delta t_2} \right]$$

$$\Delta t_{\text{atm}} = \frac{\Delta t_1 - \Delta t_2}{\ln \left[\frac{\Delta t_1}{\Delta t_2} \right]}$$

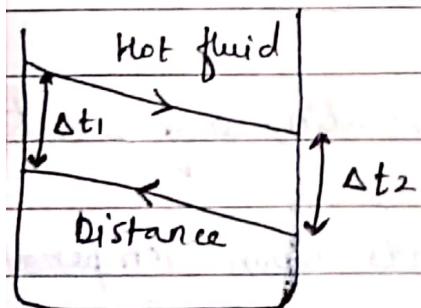
$$\ln \left[\frac{\Delta t_1}{\Delta t_2} \right]$$

$$q = VA \Delta t_{\text{atm}}$$

More will be temperature difference, more will be rate of heat transfer

Counterflow

When two liquids pass or bypass each other the type of flow is c/f counter flow.



The temp. of hot fluid at exit is lower than cold fluid at exit
 $\Delta t_1 \approx \Delta t_2$

so temp at any point of the pipe is almost same.

$$\Delta t_{\text{atm}} = \frac{\Delta t_1 + \Delta t_2}{2}$$

$$q = V \Delta t_{\text{atm}} A$$

Radiation

→ Heat transfer occurs with the help of electrostatic waves to the object.

Source	Wavelength	Application
IR (1000 °C)	1 μm	High intensity
Ceramic Rods (500 °C)	2-4 μm	

Black Body

It is defined as a body that radiates maximum possible amount of energy at a given temperature.

Rate of Radiation

→ Stefan - Boltzmann law

It gives total amount of radiation emitted by a black body -

$$q = b A T^4$$

where,

q = energy radiated per second, w (or J/s)

A = area of radiating surface, m^2

b = constant, $W/m^2 \cdot K^4$

T = absolute temperature of radiating surface, K

- It means rate of heating depends upon temperature & surface area of the emitter.
- same time, it also depends upon absorptth capacity of material to be heated.

$$b \text{ for black body} = 5.67 \times 10^{-8} \text{ } W/m^2 \cdot K^4$$

For actual bodies, as they don't radiate heat as black bodies, formula is -

$$q = \epsilon b A T^4$$

where, ϵ = emissivity of ^{actual} black body

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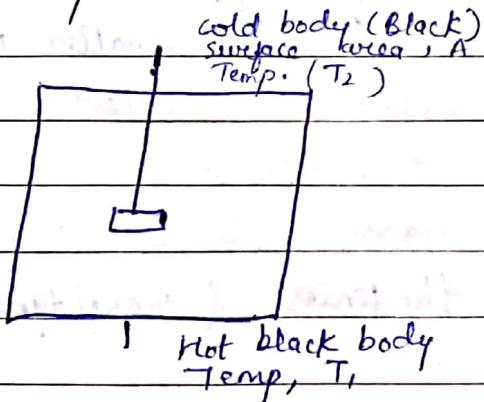
$\epsilon = \frac{\text{energy emitted by actual body}}{\text{energy emitted by black body}}$

- A good absorber of heat is also a good emitter of heat at a given temperature.
- If emissivity is equal to absorptivity ($\epsilon = \alpha$), then a substance is considered as a black body.

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Grey Bodies

→ A grey body is defined as that body whose absorptivity is constant at all wavelengths of radiation, at a given temperature.



→ consider a small cold body with a surface area of A & temp. of T_2 is completely surrounded by a hot black body at temp. T_1 .

The amount of heat transferred in such a process is expressed by Stefan Law,-

$$q = b A (T_1^4 - T_2^4)$$

This eqn assumes that all the heat radiated by a cooler body also falls on the hotter body.

Heat Exchangers & Heat Interchangers

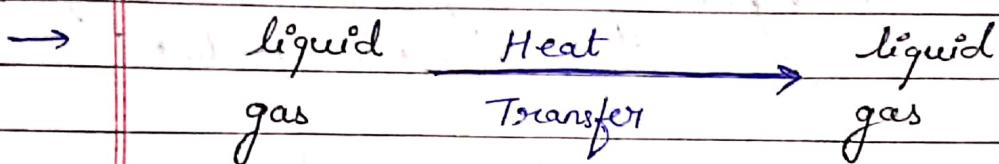
- Heat Exchangers are devices used for transferring heat from one fluid (hot gas or steam) to another fluid (liquid) through a metal wall.
- Heat Interchangers are devices used for transferring heat from one liquid to another or from one gas to another through a metal wall.

Heat Exchangers

- fluid (hot gas/steam) $\xrightarrow{\text{heat transferred}}$ another fluid (liquid)
- Equipments are :-
 - Tubular heater (shell & tube heater)
 - Multipass heater
 - two pass floating head heater.
- In this, the film coefficient on steam side are usually much larger than the film coefficient on cold liquid side.
 \therefore , overall film coefficient (heat transfer coefficient) will be nearer to cold liquid side (bcz it is smaller of the two coefficients), hence heat transfer becomes less.
- Efficiency can be improved by passing liquid at high velocity, as a result the thickness & resistance of liquid film decreases.
- Normally space inside tube is large but steam velocity is low, still they are useful, as of high value of steam film coefficient.

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Heat Exchangers



- Here, heating medium is hot liquid & the liquid to be heated is the cold liquid.
- Here film coefficients both outside & inside the tubes are of same magnitude.
- The value of overall heat transfer coefficient, U , will be near that of smaller of two film coefficient, hence heat transfer not efficient.
- film coefficient can be enhanced by inc. velocity of flow.
- from point of construct, it is difficult to inc. velo. of hot fluid outside the tubes, However, surface area contact can be increased by introducing baffles in the construction. The incd. surface area of contact enhances the coefficient. Thus rate of heat transfer is enhanced

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Baffles

- Consists of circular ~~slip~~ discs of sheet metal with one side cut way.
- These are perforated to receive tubes.
-

Definition

It is a process of vaporising large quantities of volatile liquid to get a concentrated product.

Equipments used for the evaporation are known as evaporators.

Heat is supplied to the evaporator, which transmits it to the evaporating liquid so as to provide latent heat of vaporisation.

Steam is the heating source.

* # Evaporation is surface phenomenon, i.e., mass transfer takes place from the surface.

→ No boiling occurs.

→ Surface evaporation is slow.

→ Practical definition of evaporation is the removal of solvent from the solution by boiling. The liquor in a suitable vessel f withdrawing the vapour, leaving a concentrated liquid residue in the vessel.

Difference b/w Evaporatn & other heat processesEvaporation

- Residue is conc. liquid.
- Evaporatn liqu. is only one component in most of the cases.
- No attempt is made to separate the mixture of vapour, even if any.
- The purpose of evaporation is to get conc. liquid only, but not to get crystals as it happens in some situation.

Other heat processes

- Drying:- Residue is solid.
- Distillatn:- Evaporatn liquid is combination of two or more components.
 - Distillatn:- It is compulsory to separate each component.
 - Crystallisatn:- The purpose of concentrating the sol" is to get crystals.

Applications of Evaporation

- Manufacture of bulk drugs
- Manufacture of biological products (Insulin, penicillin)
- Manufacture of Blood products (blood plasma, serum)
- Manufacture of enzymes, Hormones & antibiotics.
- Miscellaneous - water is used to get demineralised H₂O
↳ a kind of distillation.

Factors Influencing Evaporation

$$\text{mass of vap. for } \leftarrow M = K S \cdot (b - b') \rightarrow \text{press actually t in air}$$

per unit time

$\rightarrow \text{surf area of liquid exposed} \rightarrow \text{vap pres}$

$P \rightarrow \text{atm. press}$

M = mass of vapour formed per unit time (Rate), m³/s

S = surface area of liquid exposed, m²

p = atmospheric pressure, kPa

b = max. vapour pressure at the temp of air, kPa

b' = pressure due to vapour of liquid, actually present in the air, kPa

K = constant, m/s

* In general mass transfer also depends upon temperature.

• Temperature (Temp ↑, b ↑, K.E ↑ \Rightarrow molecule escape ↑)

- More temp, more will be 'b' then rate of evap. ↑.
- when temp of liquid is raised, molecules acquire sufficient K.E of escape from surface in vapour state.

→ Rate of evaporation

• Vapour Pressure (VP ↑, ROE ↑), dry air (VP↓) (ROE↑)

- VP ↑ then B.P ↓ & ROE ↑

- In presence of dry air, VP ↓ & ROE ↑

Surface Area

Surface area $\uparrow \propto$ ROE (\uparrow)

Moisture Content

- Some drug constituents undergo hydrolysis readily in presence of moisture at high temp.
- To prevent decomposn, the material is exposed to low temperature, then high temperature. ex - ^{dry extract of} belladonna.

Types of Product Required

- for conc. products \rightarrow open pan is used.
- film evaporators \rightarrow yield liquid concentrates.
- Spray dryers \rightarrow produce dry product with good solubility.
So, it decides apparatus used for evaporation.

Time of Exposure (↑ Temp & ↓ Time)

- for successful evaporatⁿ, liquid must not be heated for longer period at low temperature but it should be at high temp. & for short period.

Film & Deposit

- When vegetable extracts are concentrated in a steam pan, a film may be formed on the surface or precipitated on the heating surface.
- Film reduces the evaporatⁿ surface & precipitated matter hinders the transfer of heat. So, stirring is necessary.

Economic Factors

Economies of labour, floor space of materials are of primary considerations. The recovery of solvents & utilisation of waste heat are imp.

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Steam Jacketed Kettle or Evaporating Pan

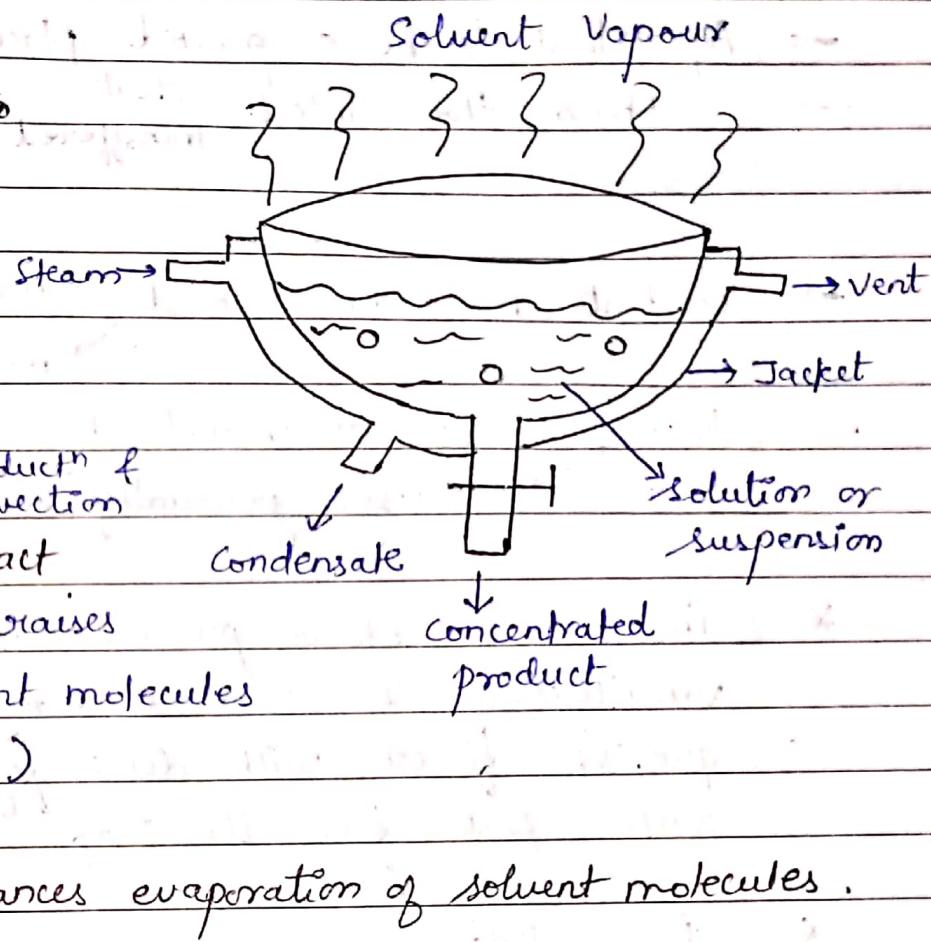
Principle

- Steam is supplied to a jacketed kettle in which ag. extract is placed.

- Steam $\xrightarrow{\text{heat}}$ kettle

heat \downarrow conduction & convection
aqueous extract

- (now temp of extract raises & escapⁿ tendency of solvent molecules into vapour increases)



Construction

- Hemispherical str. consisting of inner pan = kettle
- Inner pan enveloped with outer pan = Jacket
- Two pans are joined to enclose a space through which steam is passed.
- for smaller quantities → kettle made of single metal sheet.
- for larger capacities → several sheets are welded.
- for kettle: copper is used as ^{has} good conductivity.
- for acidic materials → tinned copper is used.
- for Jacket → Iron is used as has minimum conductivity.
- To prevent Jacket rust it is tinned or enamelled.
- Inlet for steam & outlet (vent) for noncondensed gases are provided near top of jacket.
- Condensate leaves thru outlet provided at bottom.
- kettle has an outlet for product at bottom.

Working

- Ag. extract to be evaporated placed in kettle
 - steam thru inlet $\xrightarrow{\text{Heat transferred}}$ contents
↓ condensate leaves thru outlet
 - Contents must be stirred → manually for smaller volumes
→ mechanically for larger volumes.
 - ROE → fast at initial stages
→ decreases gradually as liquid gets conc.
- * The room where process occurs must have good ventilation to remove vapours or else room is quickly filled with dense fog of condensed vapours & water falls from the roof & runs down the walls.
- * fans fitted over walls → removes vapour
→ accelerates ROE by quickly removing saturated air from the surface of liquid
- * kettle either fixed or can be tilted (90 litres) if above this (kettle wt. + content) to high to tilt it.

Uses

- for concentrating aqueous of thermostable liquors.
ex → liquorice extract

advantages → available for both large & small scale operation.
→ simple in construct, easy to operate & clean & maintain.
→ Cost of Installatⁿ & maintenance is low
→ stirring & removal of product is easy.

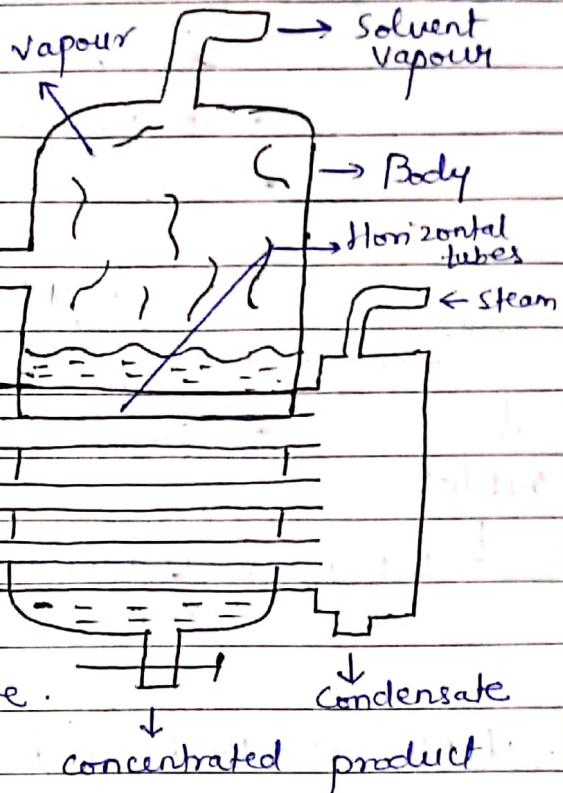
Disadv → • Heat economy is less, hence cost per unit material produc.

- Not suitable for heat sensitive materials, ∵ long exposure.
- Heatⁿ area ↓ as product gets more concentrated.
- Saturatⁿ may occur due to its open type ppt.
- BP of the can't be reduced, ∵ pressure can't be created in open.

Horizontal Tube Evaporator

Principle

→ Steam is passed through horizontal tubes, which are immersed in a pool of liquid to be evaporated.



→ Heat transfer takes place through tubes & liquid outside tubes get heated.

→ Solvent gets evaporated & escapes from top of evaporator.

→ Concentrated liquid collected from the bottom.

Construction

→ Large cylindrical body with conical or dome-shaped top & bottom, made up of cast iron or plate steel.

→ Avg. size ranges from 1.8 to 2.4 meters in diameter & 2.4 to 3.6 meters height.

→ Lower part of body has steam compartment with an steam inlet at one end & a vent for non-condensed gases on ^{other} end.

→ Condensate outlet provided at bottom of steam compartment.

→ In steam compartment, 6-8 stainless steel horizontal tubes fit.

→ Tubes cut long enough so that they project about 25 mm beyond tube sheet on both ends.

→ Width of steam compartment is usually half the diameter of body.

→ At one convenient point, an inlet for feed is provided.

→ One outlet for vapour provided at top of dome.

→ Another outlet for thick liquid is placed at centre of the conical bottom of body.

- Working -
- feed introduced into evaporator until steam compartment is satisfactorily immersed
 - steam \rightarrow steam compartment
| Heat

Horizontal tubes
 escapes heat &
 evaporated \leftarrow gets liquid
 | Heat, co of temp. gradient
 outlet

- Steam condensate pass thru correspond' outlet.

\hookrightarrow process continues till a thick lig is formed & is collected from bottom outlet.

Uses \rightarrow Best used for (non-viscous solutions) that do not deposit scales or crystals on evaporation.
 ex \rightarrow Cascara extract.

Adv \rightarrow cost per sq. meter of heat' surface is usually less.

Climbing Film (Rising Film) Evaporator

Principle

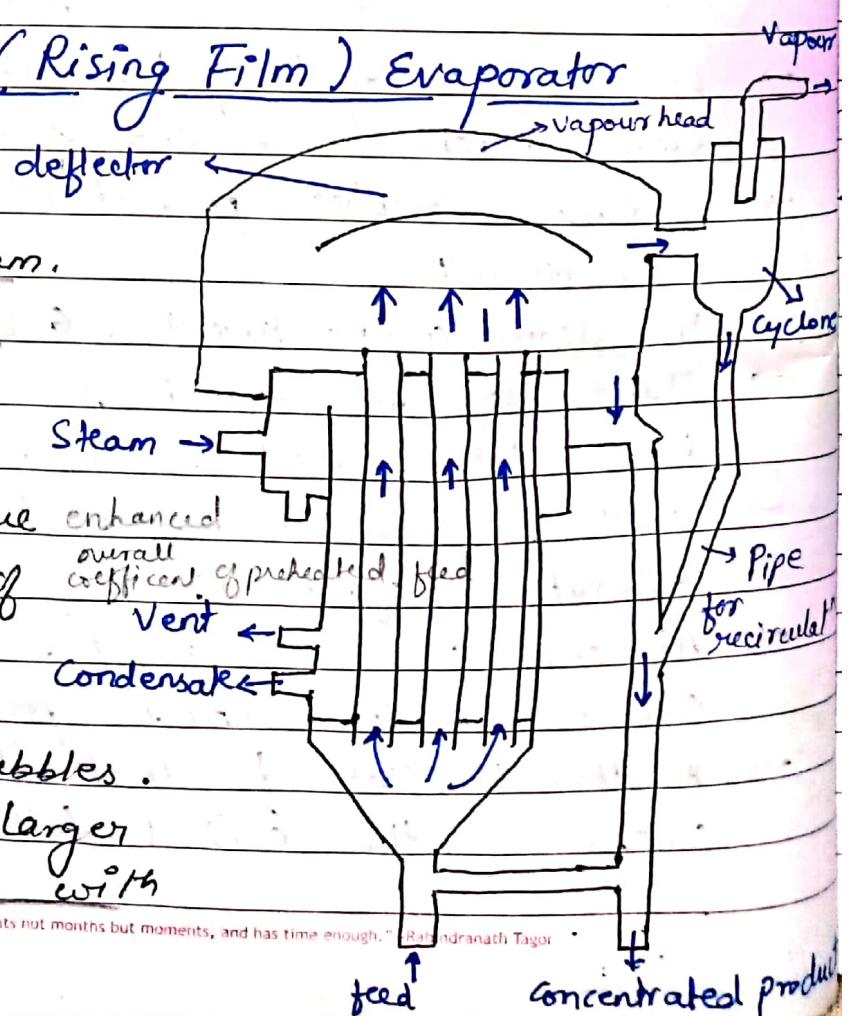
Tubes heated externally by steam.

Preheated ^{feed} steam enters from bottom & flows up thru heated tubes.

Liquid rapidly gets heated due enhanced overall coefficient of preheated feed.

The liquid near walls becomes vapour & forms small bubbles.

These tends to fuse to form larger bubbles, which travel up tubes with entrapped slug.



"The butterfly counts not months but moments, and has time enough." Rabindranath Tagore

→ liquid films are blown up from top of tube & strikes entrainment separator (deflector), it throws the conc lig down in lower part & from there it is withdrawn

Construction

- Heating unit has steam jacketed tubes.
- Tubes (long & narrow) held b/w two plates.
- An entrainment separator at top to vapour end.
- Evaporator carries steam inlet, vent & condensate outlet.
- feed inlet is from the bottom of steam compartment.

Working

→ Preheated lig. feed introduced to bottom of unit.
→ Height of lig. column maintained low, i.e., 0.6 or 1.2 m above the bottom tube sheet.

- Steam enters into spaces outside the tube three inlet.
- Walls of tube $\xrightarrow{\text{heat}}$ liquor $\xrightarrow{\text{forms}}$ vapour $\xrightarrow{\text{forms}}$ small bubbles

as more vapour is formed, the slug of lig. is blown up in the tubes facilitating the liquid to spread over the walls as film
 \downarrow

It vapourise continuously & rapidly \longrightarrow liquid concentrate + vapour eject at \uparrow velo. from top of tube

- * Entrainment separator not only prevents entrainment, but also acts as a foam breaker.
- * Vapours leave from top while concentrate is collected from bottom.

- Uses → for thermolabile substances (insulin, liver extracts of vitamins) are more concentrated.
- Clear, foaming of corrosive liquids can be operated.
- Deposits of scale can be removed quickly by raising feed rate or reducing steam rate so that product is unsaturated for a short time.

- Advantages →
- Large area for heat transfer is provided (long narrow tube)
 - Due to ↑ velocity of liq., resistance for heat transfer is reduced.
 - Suitable for heat sensitive materials as time of contact b/w liquid & heat surface is very short.
 - Suitable for foam forming liquids.
 - If requires low hold up small floor space.
 - No elevatn is bp due to hydrostatic head.

- Disadv
- Expensive & complicated construction.
 - Difficult to clean & maintain
 - Large head space required.
 - Not advisable for very viscous liquids, salt & scaling liq.

Forced Circulation Evaporator

Principle

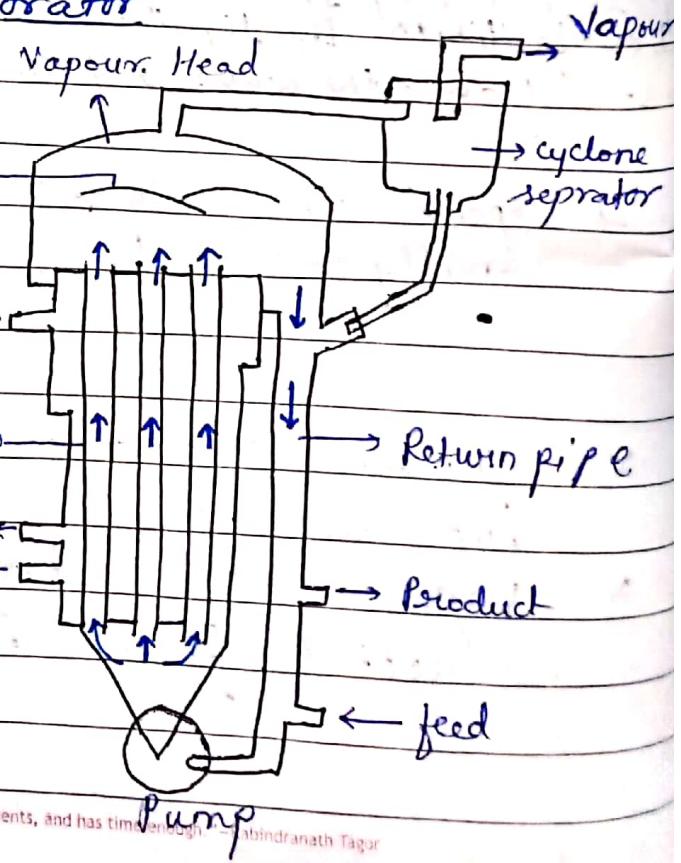
Liquid is circulated thru the Deflector tubes at ↑ press. by means of a pump.

Hence, boiling does not occur & steam → Bp is elevated.

Forced circulat' also creates Heating Tubes some form of agitation.

when liquid leaves the tubes Condensate enters the Vapour head, pressure falls suddenly. This leads to flashing of super heated liquor.

Thus evap. is affected.



The butterfly requires not months but moments, and has time enough. Rabindranath Tagore

Constructⁿ

- Steam jacketed tubes are held b/w 2 tube sheets.
- Tube measures 0.1m in diameter & 2.5m long.
- ^h Flash chamber (tube part projects vapour head), has a deflector.
- Vapour head connected to a return pipe, which runs downwards & enters into inlet of pump.

Working

- Steam → Calandria
 - Pump → liquid → tubes with + velocity.
 - liquid moves up to tube → gets heated & boils.
 - Vapour + liquid → rush out of tube at ↑ velocity.
↓
strikes deflector → throws liquid downwards
effective separn of liq & vapour.
- ✓ enters cyclone separator & leaves equipment.
returns to pump for further evaportn
Conc. product collected.

- ### Uses
- at + pressure, useful for thermolabile substances.
 - conc' of insulin & liver extracts.
 - useful for crystallizing operators.

Advantages → Heat transfer coefficient is high due to rapid liq movement.

- → Saltⁿ, scalⁿ & fouling not psbl
- → Suitable for thermolabile products.
- → Suitable for high viscous prep. as pump mech. used.

Disadv.

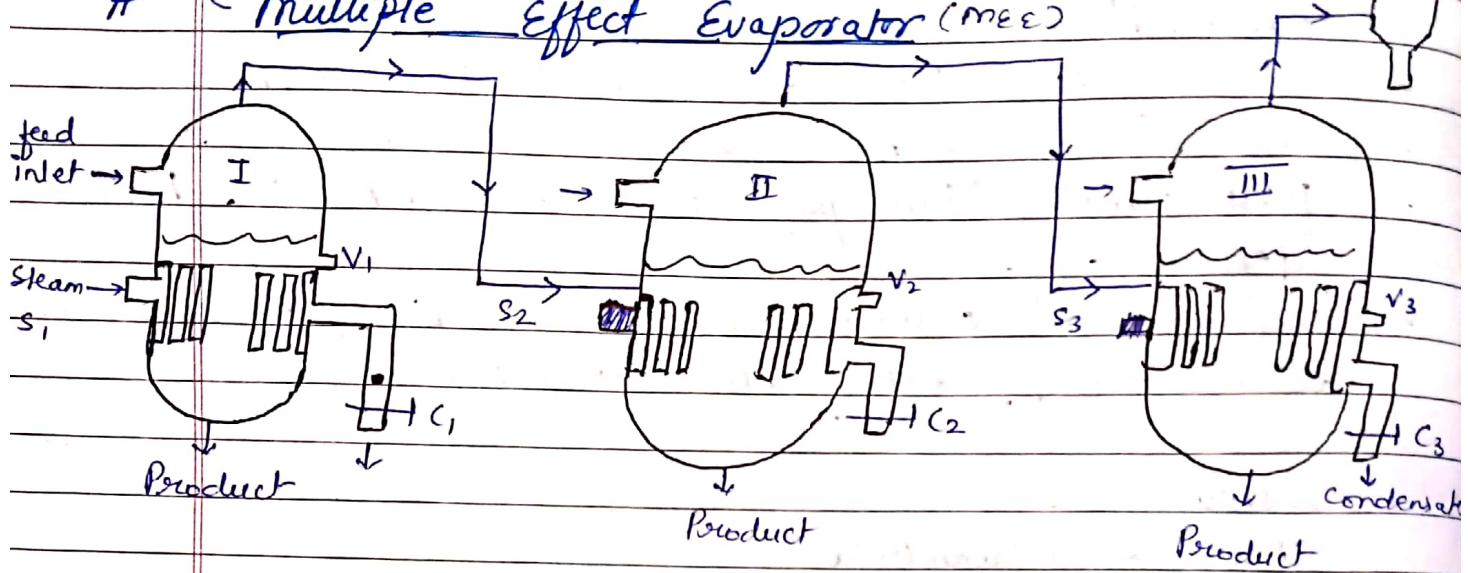
- Holdup of liq. is high.
- Expensive as power pump req. for circulatn the liq.

when vertical tube evaporators are connected in several ways so to achieve large scale evaporation as well as greater economy, is called MEE.

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multiple Effect Evaporator (MEE)



Advantages

- Suitable for large scale of continuous operations.
- ↑ economic value when compared to single effect.
- 5 evaporators can be attached.

Construction

- Same as vertical tube evaporator.
- Vapour from 1st evaporator serves as a heating medium for the 2nd & similarly of 2nd for 3rd.

Working

- Hot saturated soln of feed is directly fed to each of three effects in parallel w/o transfer of material from one effect to other effect.
- In beginning, equipment is at room temp & atm. pressure.
- Liquid feed is introduced to all 3 evaporators upto level of upper tube sheets, then -
- Vent valves V_1 , V_2 & V_3 are open & all other are closed.
- ↑ vacuum is created in lig. chambers of evaporators.
- S_1 & C_1 are opened, steam is supplied.
- Steam replaces cold air in 1st evaporator & when cold air is removed, V_1 is closed.

"The butterfly cannot fly moments but moments, and has time enough" — Rabindranath Tagore

- Steam is continued till desired pressure P_0 is created in the steam space of 1st evaporator & gets condensed. Condensate is removed thru C_1 .
- Due to heat transfer, lig. temp \uparrow & reaches the BP. Vapour will be generated from the feed.
- Vapours displaces air in upper part of 1st evaporators. Vapours also displaces steam space in 2nd evaporator.
- After displacement of air by vapours in steam compartment of 2nd evaporator, V_2 gets closed.
- Vapours of 1st evaporator transmits its heat to the liquid of 2nd evaporator & gets condensed. Condensate removed thru C_2 .
- Steps continued in 3rd evaporator.

Economy of Multiple Effect Evaporator

Economy of an evaporator is the quantity of vapour produced per unit steam emitted.

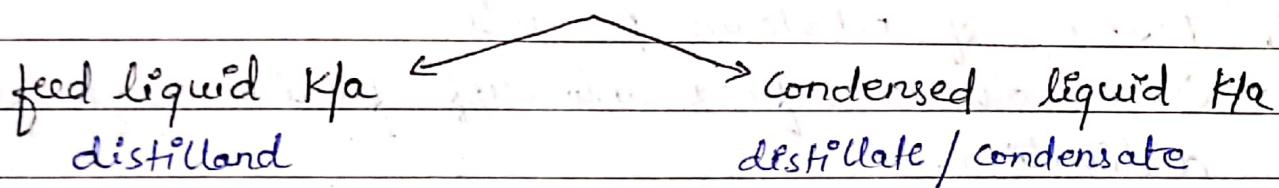
$$\rightarrow \text{Economy} = \frac{\text{Total Mass of Vapour Produced}}{\text{Total mass of steam supplied}}$$

$$\rightarrow \text{Single effect} = \frac{N \text{ no. of mass of Vapour}}{N \text{ no. of mass of steam}} = 1$$

$$\rightarrow \text{Multiple Effect} = \frac{N \text{ no. of vapour}}{1} = N$$

Distillation

- It is defined as the separation of components of a liquid mixture by a process involving vaporization & subsequent condensation at other place.
- This process involves 2 steps:-
- Converting a liquid into vapour phase
 - Transferring the vapours to another place & recovering the liquid by condensation.



- * It is used to separate liquids if one is volatile & another is non-volatile liquid.
 - * Volatile + Non-volatile liquid
 - Heat → Heat (does not vaporise easily)
 - evaporates readily
 - (On condensatn, major product)
- ∴, distillation is said to be partial separation method.

Diff. b/w

- Distillatn - when condensed vapour is required.
- Evaporatn - when concentrated liquid residue is required, temp is maintained below B.P. of liquid, further, vapour is not condensed until recovery is essential.
- Drying - when dried solid residue is required.

Applications

(1)

separation of volatile oils

Volatile oils are separated from cloves, anise seeds & eucalyptus leaves by simple distillation.

(2)

Purification of organic solvents

- Normally simple distillation is used, it is also used for determining the boiling range of liq. as per IP.
- Absolute alcohol (100% ethanol) obtained by azeotropic distillatn.

(3)

Refining of petroleum products

- Crude oil is refined into diff. fractions using flash distillation.
- Each fraction is a multicomponent system.
ex → Petroleum ether 60, 80, etc

(4)

Recovery of Solvents

Solvents used for extractn of drugs from plant parts, these solvents must be recovered to avoid environmental contamination

(5)

Quality Control methods

Used for determining alcohol content in liq. dosage forms such as elixirs, as per IP, 1996.

(6)

Separation of drugs obtained from plant & animal sources

→ Vitamin A separated from fish liver oil using the method of molecular distillation.

(7)

Purification of drugs obtained from chemical process

#

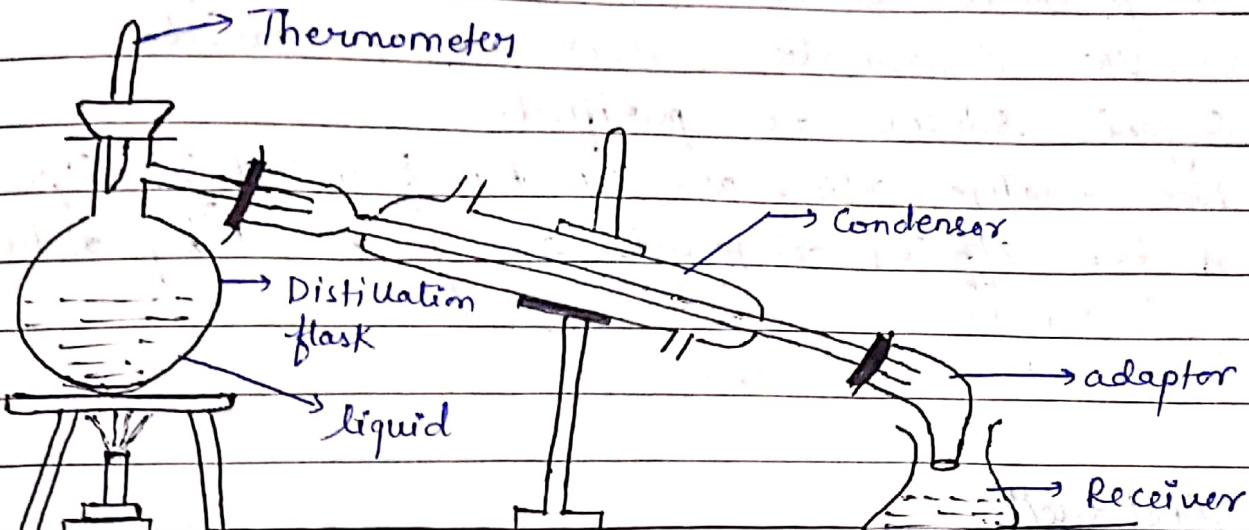
Classification of Distillation

- Simple distillation ✓
- flash distillation ✓
- Fractional distillation ✓
- Azeotropic & extractive distillation
- Distillation under reduced pressure. ✓
- Steam distillation ✓
- molecular distillation ✓

"The butterfly counts not months but moments, and has time enough." —Rabindranath Tagore

#

Simple distillation (SD)



- Simple distillation is a process of converting a single constituent from a liquid (or mixture) into its vapour, transferring the vapour to another place of recovering the liquid by condensing the vapour, usually by allowing it to come in contact with a cold surface.
- This process is also known differential distillation, as distillation is based on the differences in volatilities & vapour pressures of the components in the mixture.

Principle

- Liquid boils when V.P of liq = Atmospheric pressure.
- SD is conducted at its boiling point.
- ↑ the relative volatility of liquid, better is the separation by simple distillation.
- Heat is supplied to the liquid so that it boils.
- Resulting vapour transferred to a diff. place & condensed.
- If the liquid of interest is volatile & remaining components are nonvolatile, then SD is useful means of purificat' & separat' of liquids.

Applications

- To prepare distilled water & water for injection.
- Volatile & aromatic waters are prepared.
- Organic solvents are purified.
- Non-volatile solids are separated from volatile liquids.
- A few official compounds are prepared by distillation.
ex →
 - spirit of nitrous ether
 - Aromatic spirit of ammonia

Construction

- A distillation flask with a side arm sloping downwards.
- Condenser fitted to side arm by means of a cork.
- Condenser is usually water condenser, i.e., jacketed for circulation of water.
- The condenser is connected to a receiver flask using an adapter.
- On laboratory scale, whole apparatus made up of glass.

Procedure

- liquid → flask → bumping avoided by porcelain pieces or porous pot before distilltn
- Thermometer fitted in flask with help of cork.
- Thermometer bulb is just below the level of side arm.
- water circulated thru jacket of condenser.
- Contents → heated gradually → liquid boils

Temp of distillate ← Temp rises ← Vapour rises up &
is noted down & reaches to passes down the side arm
if is eq. to B.P a const. value to condensor.

* flame adjusted so that distillate Condensed & Collected to
is collected at the rate of 1-2 drops/sec. receiver
* If is continued until small amount of lig. remains in flask.

"The butterfly counts not months but moments, and has time enough." Rabindranath Tagore

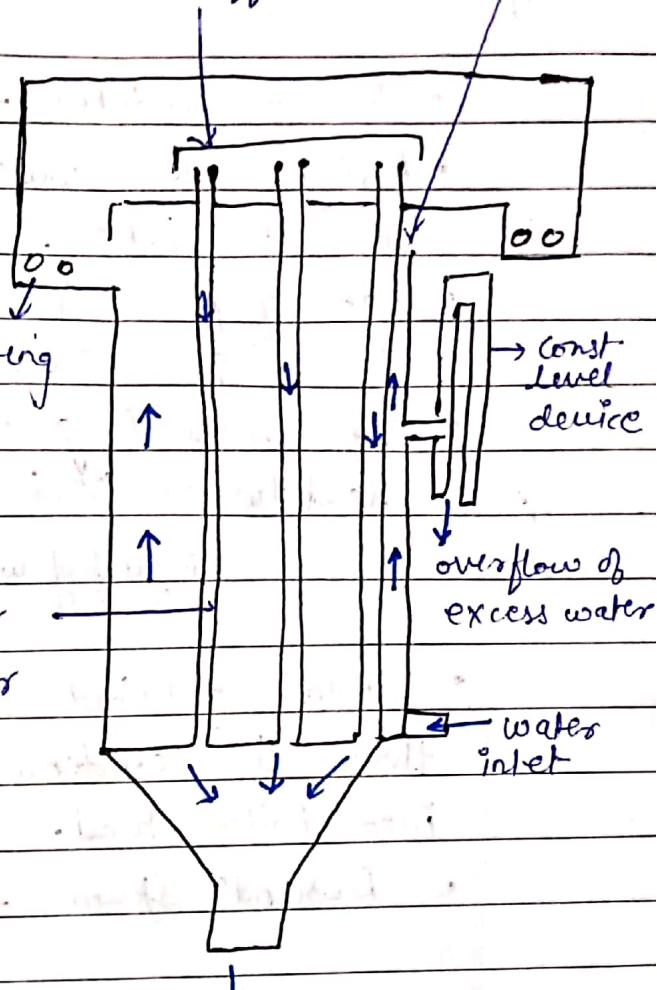
Water For Injection

Baffles

Escape of Gase

Principle Same as that of SD, however it is special case for following reasons —

- Gases dissolved in the raw water must be removed.
- These should not be allowed to contaminate the distillate, gases include CO_2 & NH_3 . Multi-tubular condenser most imp to remove.
- Carry over of soluble materials in the droplets must be avoided mainly when it is used for WOI.
- Entrapment of liquid droplets by the vapour must be prevented. for this purpose, baffles are included in path of vapour b/w boiler & condensor.
- Contaminatn of distillate by pyrogen from feed water must be avoided.
- The residue of solids must not be concentrated to a point where hydrolysis occurs. Otherwise, distillate may be contaminated by volatile material produced during hydrolysis.
ex → Hydrolysis of chlorides, produce HCl .



Construction

- Consists of a boiler, made up of cast iron.
- Baffles of condenser tubes made up of stainless steel or monel metal
- Top of condenser jacket is open, so that gases from water can escape to atmosphere.
- Condenser tubes are vertical & open at both ends.

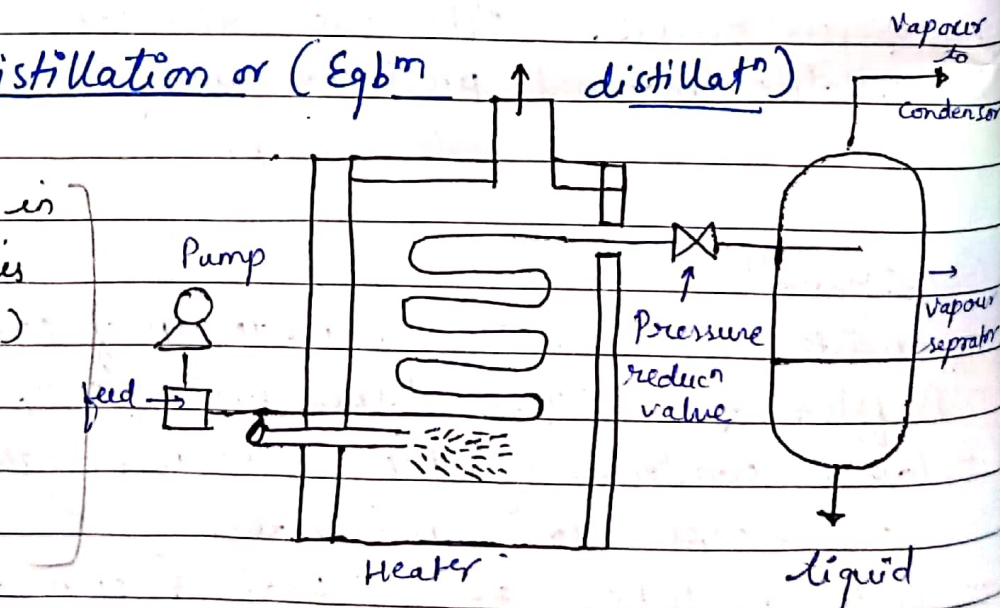
- Working -
- water (feed) \rightarrow base of still \rightarrow rises to jacket (has no of tubes)
 - In condenser tubes \rightarrow condensed ^{liquid} tubes descends
 - Rising feed water gets heated on account of condensate in the tubes.
 - Rate of flow is adjusted that water gets heated to $90 - 95^\circ\text{C}$, before it enters the boiler.
 - The dissolved gases in water escape to atmosphere.
 - Heated water $\xrightarrow{\text{enters}}$ boilers
 \quad (steam is circulated under press. thru coil)
 - Steam obtained thru feed water can't escape except thru the condenser tubes, whose upper ends are protruded into boiler head.
 - Descendⁿ steam \rightarrow condensed into distilled water
- \downarrow
- flows from lower end of tube

Advantages

- economical, as amount of steam used is reduced, preheated feed is used by counter-current flow of condensate.
- Also facilitates escape of dissolved gases w/o any additional efforts.

Flash Distillation or (Egbm. \uparrow distillatⁿ)

It is defined as process in which entire liquid mix. is suddenly vapourised (flash) by passing the feed from ↑ pressure zone to low pressure zone.



"The butterfly counts not months but moments, and has time enough." — Rabindranath Tagore

- Also cld as equilibrium distillation, i.e., separation is attempted when liquid & vapour phases are in eqb^m.

Principle

- Hot liquid \rightarrow entered from \rightarrow \uparrow pressure zone to

Flash Vapourisation = entire liquid mix. \leftarrow suddenly vaporize \downarrow pressure zone

- Durin process \rightarrow chamber \rightarrow gets cooled.
- Individual vapour phase molecules of \uparrow boiling fraction gets condensed, while \downarrow boiling fractions remain as vapour.
- Process requires certain amount of time.
 \therefore liq. & vapour kept in intimate contact until eqb^m is achieved.
- Liquid fractⁿ collected separately, vapour is separated from liq. & further allowed to condense.

construction

- Consists of pump \rightarrow connected to \rightarrow feed reservoir.
- Pump helps feed to \rightarrow Heatⁿ chamber which has heatⁿ mechanis^m.
- Other end of pipe directly introduced into vapour-liquid separator thru a reduce value.
- Vapour outlet at top of separator & liq. outlet at bottom.

- work -
- feed pumped thru heater at a certain pressure.
 - liq \rightarrow heated \rightarrow enters vapour-liq separator thru pressure reduce values.
 - Due to pressure drop \rightarrow hot liq flashes \rightarrow vaporisat^m tendencies

↓
cooling \leftarrow sudden vaporisat^m

- Individual vapour phase molecules of \uparrow Boiling fractⁿ gets condensed, low Boilⁿ fractⁿ = as vapour
- mixture given time so as vapour & liquid separates & eqb^m is achieved.

"Faith is the bird that feels the light when the dawn is still dark." — Rabindranath Tagore

- Vapour separated \rightarrow pipe above liquid " \rightarrow pipe at bottom of separator
- Continuously instilling feed, continuous flash distil. can be obtained.
- Operate condtn adjusted such that, amount of feed = amount of material removed.
 ↓
 i. Vapour & liq. conc at any point remains constant.

Uses

- To separate components with wide temp. diff.
- Widely used in petroleum industry for refining crude oil.

Adv -

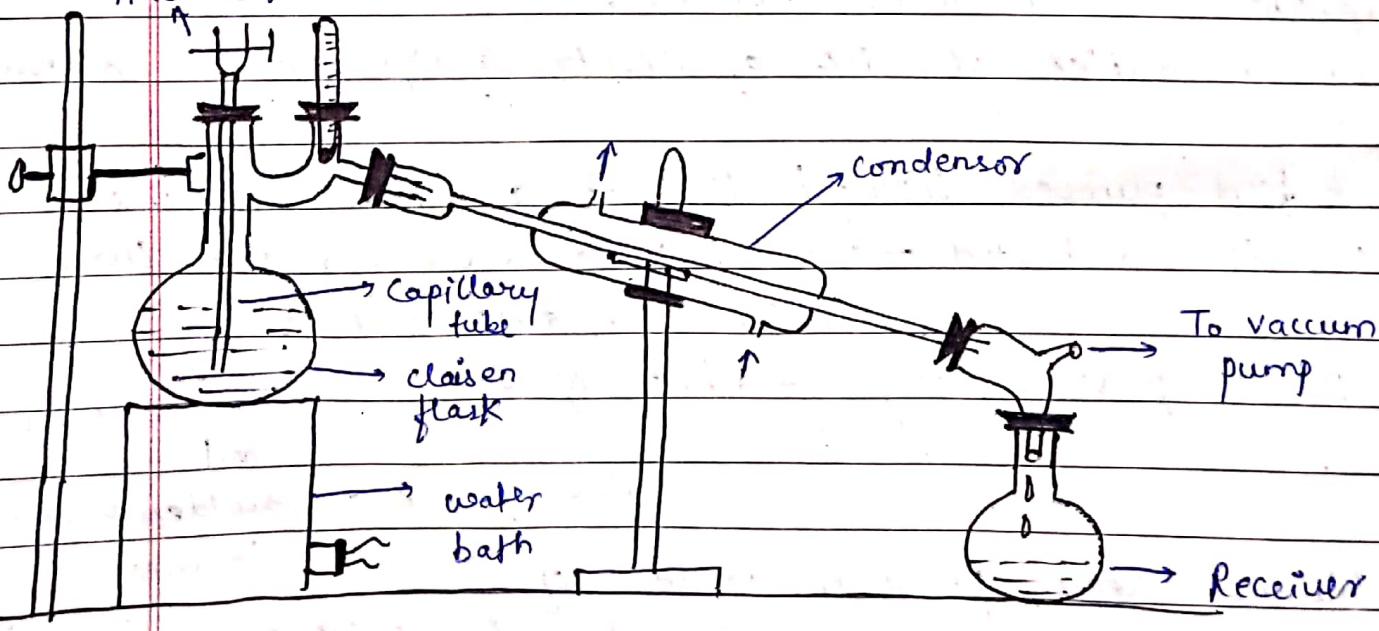
- Continuous process
- Used to obtain multi-component system of narrow boiling range (especially in oil refinery).

Disad -

- Not useful to separate components of comparable volatility.
- Not useful for 2 component system.
- Can't get pure components.

Distillation under reduced pressure

Prin. cock



Definition

- Defined as distillation process in which the liquid is distilled at a temp lower than its B.P by the application of vacuum.
- Vacuum, such pumps are used to reduce press of lig. surface.

• Mass of Vapour formed \propto VP \propto 1

atm pressure

- When atm pressure is reduced by applying vacuum, the BP of lig is reduced as lig. boils at lower temp than its BP.

Principle → • VP of lig = Atm V.P, i.e. pres. on its surface \Rightarrow lig. boils

- Ext. pres. \downarrow by vacuum, B.P also \downarrow

ex → water = B.P at 100°C at 101.31 kPa

at 40°C V.P = 9.33 kPa , Now $(\frac{\text{BP}}{\text{V.P}})_{\text{H}_2\text{O}} = 40^\circ\text{C}$

∴ Net result = \uparrow in rate of mass transfer into vapour.

→ acc. to this, water at 40°C , 9.33 kPa boils, mass of vap. formed in unit time is approx 11 times, i.e., ratio $760 / 70$ for H_2O .

Construction - double neck distillat' flask = claisen flask

- Thick walled glass apparatus with interchangeable stand. glass joints are used for vacc. distillat'.
- Claisen flask \rightarrow one neck \rightarrow Thermometer
 - ↳ other neck \rightarrow prevents splash of violently agitated lig.
- Bump' occurs, thus can be avoided by plac' fine capillary.
- Capillary tube is dipped in boiling lig, so that stream of air bubbles is drawn out.
- Water/oil bath used for heat.
- Claisen flask \rightarrow condenser \rightarrow receiver.
- Vacuum pump attached to adapter to receiver.

a vacuum gauge placed b/w vacuum pump & receiver

"Faith is the bird that feels the light when the dawn is still dark." — Rabindranath Tagore

Working

→ liquid → flask + Porcelain
 (1/2 or 2/3)
 (facilitate distill)
 (Avoids Bumps)

→ Required vacuum is applied → contents heated gradual

Condensate ← Vapour pass ← Temp ↑ of lig vapour
 collected in receiver thru condensor due to vacuum

- Temp noted down which is < BP of liquid
- Convenient is to distill small quantities at a time.

Applications -

- (1) Preventing degradation of active constituents

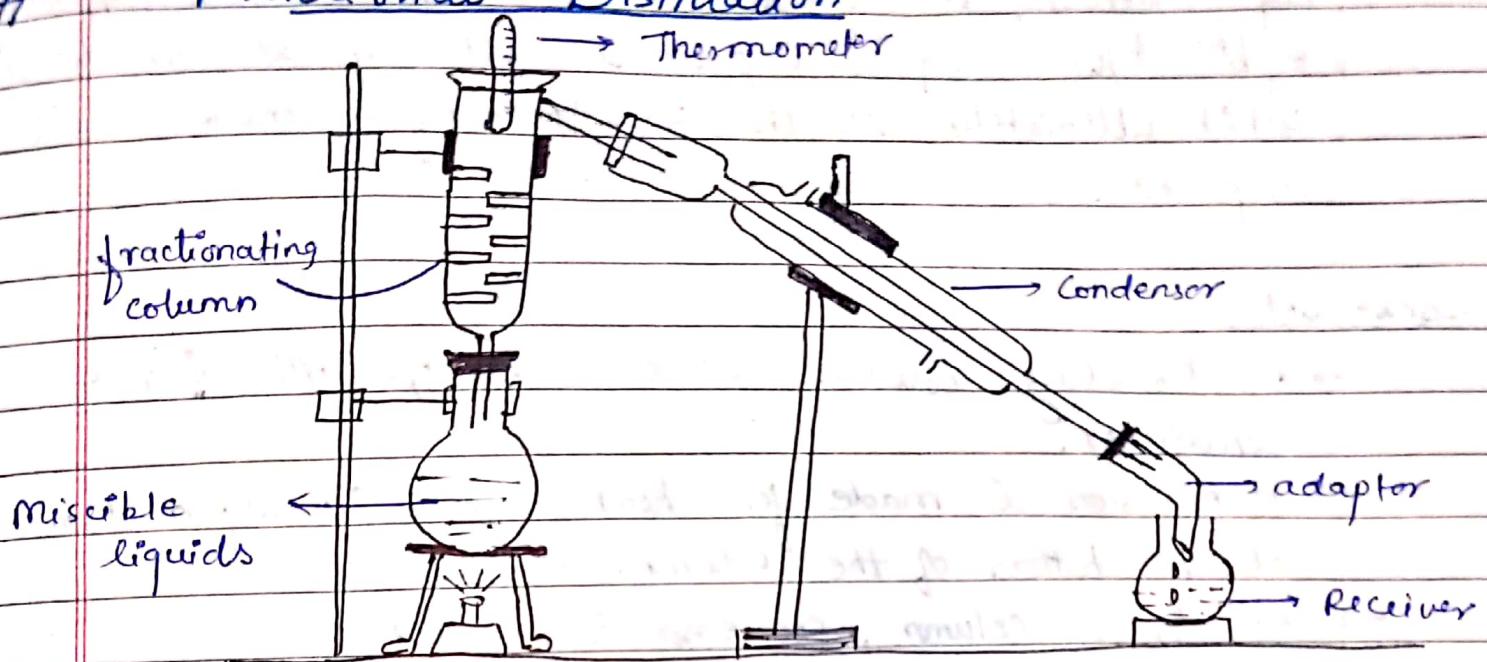
Category	Reaction	Examples
Enzyme	Inactivation	malt extract, pancreatin
Vitamins	Oxidation	thiamine, ascorbic acid
Glycosides	Hydrolysis	anthraquinones
Alkaloids	Racemization	hyoscyamine to atropine
Tannins	Precipitation	phlobatannins to phlobaphenes

- (2) Changing physical form

To prepare cascara sagrada tablets, gomual extract is suitable.

- Disad → . Persistent foaming occurs, can be overcome by addⁿ caprylic alcohol or by insertⁿ fine capillary.
 • Not suitable for preparing semisolid or solid extracts.

Fractional Distillation



→ It is a process in which vaporisation of liquid mixture gives rise to a mix. of constituents from which the desired one is separated in pure form.

→ This method is ~~not~~ a rectification, as a part of vapour is condensed & returned as a liquid.

Used to separate miscible volatile liq., whose BP are close, by means of fractionating columns.

* In SD → Vapour directly passes to condenser

* In FD → Vapour must pass thru fractionating column in which partial condensation of vapour is allowed to occur, a part of condensing vapour returns to still.

Principle

- A mass transfer process involving counter-current diffusion of the components at each eqbm stage.
- In distillatn, partial condensatn of vapour is allowed to occur in fractionating column.
- In column, ascendⁿ vapour from still is allowed to come in contact with the condensing vapour returning to the still.
- This results in enrichment of vapour with the more volatile component.

→ By condensing the vapour & reheating the liq. repeatedly eq b/w b/w liq & vapour is set up at each stage, which ultimately results in sep. of a more volatile component.

Construction

- fractionating column is inserted b/w still & the condenser.
- A provision is made for heat supply (usually a steam coil) at the bottom of the column.
- At top of column, condenser is provided.
- Column has a large area for providing sufficient flow conditions.
- The broken lines across the column represent the contacting devices.

Working

- 1. mixture → Boiler & heated usually by steam
 - To^w miscible liq. A & B containing 20% A & 80% B.
 - Liq. A (more volatile component, MVC) & liq B (LVC)
 - These liq. don't produce const. B.P mixture

* Graph illustrated afterwards →

Theory

- acc. to principle of colligative pts, when a substance is dissolved in a liquid, the VP of solvent is lowered.
- 2 miscible liquids are mixed, each may be considered as a solution of one in other, VP of each component is lowered, pressure exerted by each one is p/a partial pressure.
- Acc. to Dalton's law, total pressure exerted by a gaseous mix. is the sum of the individual partial

pressures of the component gases.

$A \& B \rightarrow$ 2 miscible liquids

$P_A \& P_B \rightarrow$ their partial pressures, Dalton's Law \rightarrow

$$\text{Total pressure} = P_A + P_B$$

- Dalton's law imp. cz it permits estimation of total VP, which should be equal to atm. press. so as to reach BP.

At BP, vapor. is max.

To condenser

- Holds when partial pressure are not too high.

Graph

1) when the BP of mix is reached, the vapour compostn curves are drawn as shown by lowest pair of curves.

These curves indicate that the vapour contains 60% of A

2) when this vapour is condensed, the resulting liq. is again heated to BP, this vapour gives the composition containing 90% of A (2nd pair of curves) from the bottom

3) This vapour impinging on a cool surface & gets condensed.

This fraction is re-vapourised by heating to its boiling point.

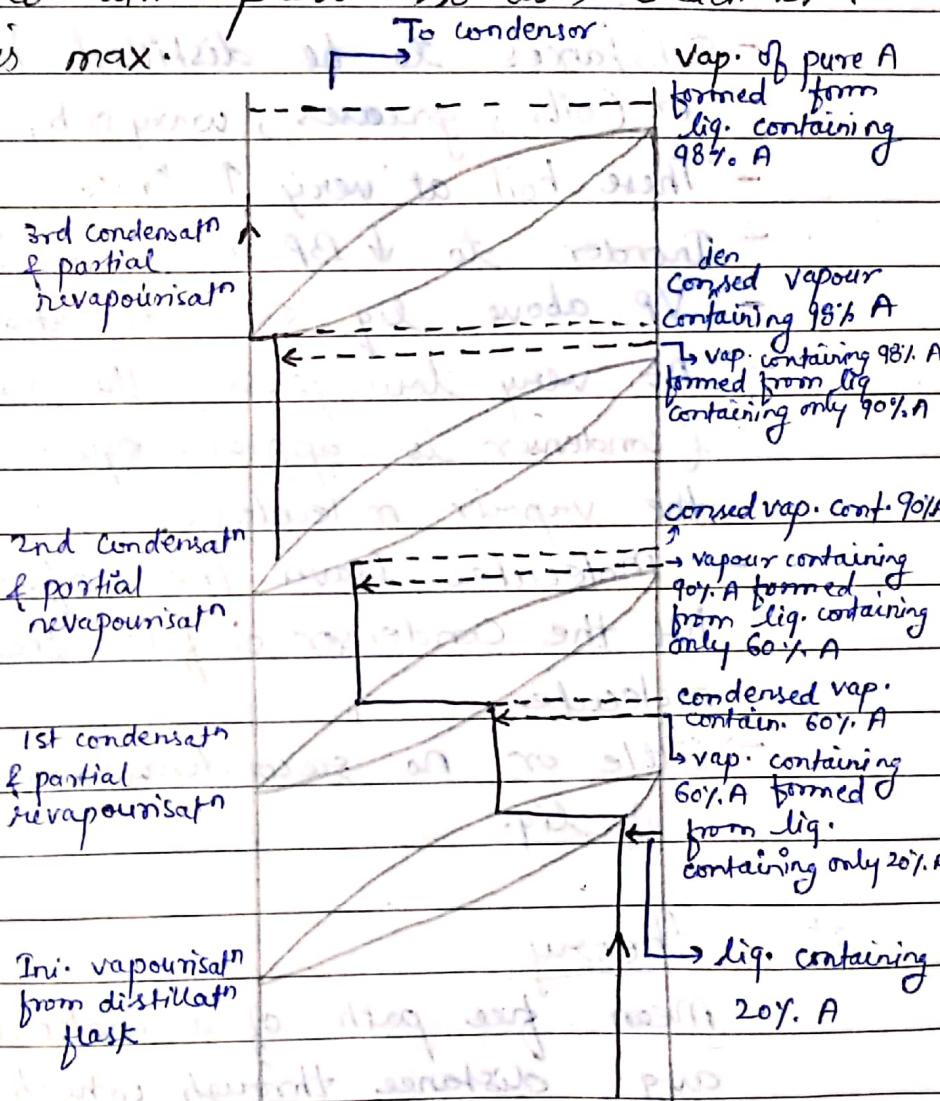
This BP curve of

this distillate 98% of A

(3rd pair of curves from the bottom)

4) This fraction of vapour impinges on a cooling surface. This gives fourth pair of curves. Now vapour contains higher (more than 98%) proportion of A i.e., vapour of pure component.

5) The vapour moves to a condenser at the top of column & gets condensed.



#

Molecular Distillation

- Defined as distillation process in which each molecule in the vapour phase travels mean free path if gets condensed individually w/o intermolecular collisions on application of vacuum.
- Also c/d as Evaporative distillation or short path distillation

Principle

- Substances to be distilled have very low vap. pressure ex (oils, greases, waxy sub., high mol. wt. sub.)
- These boil at very \uparrow temp.
- In order to \downarrow BP of liq., \uparrow vacuum must be applied.
- VP above liq. << saturated vapour \rightarrow In eqbm.
- At very low press., the dist b/w evaporate surface & condenser is approx. equal to mean free path of the vapour molecules.
- Molecules leave the surface of liq. are more likely hit the condenser surface than to collide with other molecule.
- Little or no recondensation takes place at surface of liq.

#

Theory

Mean free path of a molecule is defined as the avg. distance through which a mol. can move w/o coming into collision with other.

$$\lambda = \frac{\eta}{\rho f} \sqrt{\frac{3}{P}}$$

ρ = vapour press, kPa

f = density, kg/m³

η = viscosity, Pa.s

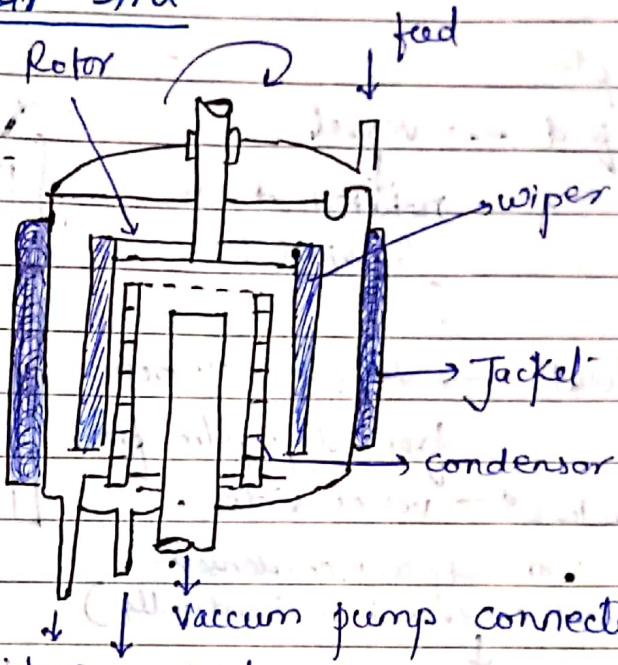
λ = mean path length, m

"The butterfly counts not months, but moments, and has time enough." - Rabindranath Tagore

Falling film Molecular still or Wiped Film molecular still

Principle

- Vap. occur from a film of liquid flow down a heated surface under \uparrow vacuum.
- The vapour (molecules) travels a short distance of strikes the condenser nearby.
- Each molecule is condensed individually.
- The distillate is subsequently collected. Residue product



Construction

- Vessel has diameter = 1 m.
- Vessel walls have suitable heating means (jacket).
- Wipers provided adjacent to vessel walls & connected to a rotatⁿ head thru a rotor.
- Condensor arranged very close to wall (evaporatⁿ surface).
- Vacuum pump connected to large diameter pipe at centre of vessel.
- Provision made for collecting distillate & undistilled lig residue at the bottom.

Working

Vessel \rightarrow heated, Vacuum applied \rightarrow centre of vessels, wipers \rightarrow rotate

feed \rightarrow Inlet \rightarrow liquid flows down the walls \rightarrow forms film by PTFE wipers

condensate \leftarrow Vapour \leftarrow \therefore surface already
collected travels its heated film \leftarrow • (wipers move at 3 m/s)
in vessel mean free path of strike evaporates directly \leftarrow • (film velocity \approx 1.5 m/s)

condenser

↓

residue collected from bottom of vessel \rightarrow re-circulated

* capacity = 1000 l/Hour

"Faith is the bird that feeds the soul when the dawn is still dark." - Rabindranath Tagore

Centrifugal Molecular Still

Principle

- lq. feed \rightarrow vessel

↓
rotated at very
↑ speed

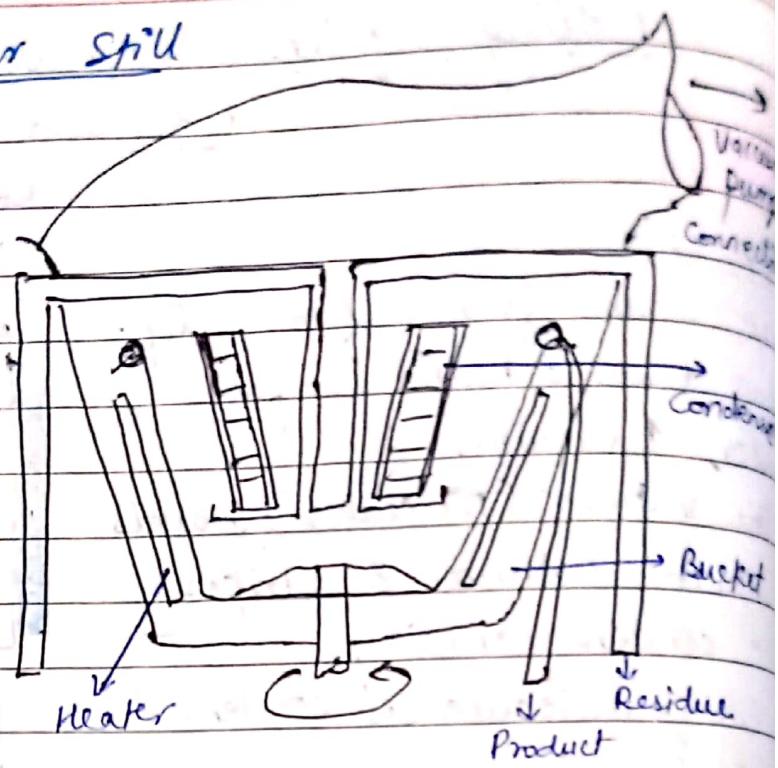
(centrifugal action)

- on heatn \rightarrow vapourisatn occur

Travel
short distance \leftarrow vessel side.

f condense on adjacent condensers
(each mol. condensed individually)

↓
distillate collected.



Construction

- Bucket shaped vessel \rightarrow dia = 1 to 1.5m
- rotated ↑ speed with help of rotor (motor)
- Radiant heaters are provided externally to heat fluid in bucket.
- Condensers arranged very close to evaportn surface.
- Vacuum pump connected to entire vessel at top.
- To introduce feed provisions are made into centre of bucket;
also to receive residue f for recirculatn.

Work

• Vacuum \rightarrow centre of vessel \rightarrow rotated at ↑ speed

• feed \rightarrow centre of vessel \rightarrow due to centrifugal action

lq. moves outwards over the
surface of vessel & forms film.

strikes \leftarrow vapours

Condenser travel mean free path \leftarrow radiant heaters heated surface
evaportn occurs from film.

Collected
to another
vessel.

residue collected

from vessel bottom
& is recirculated
then feed port.

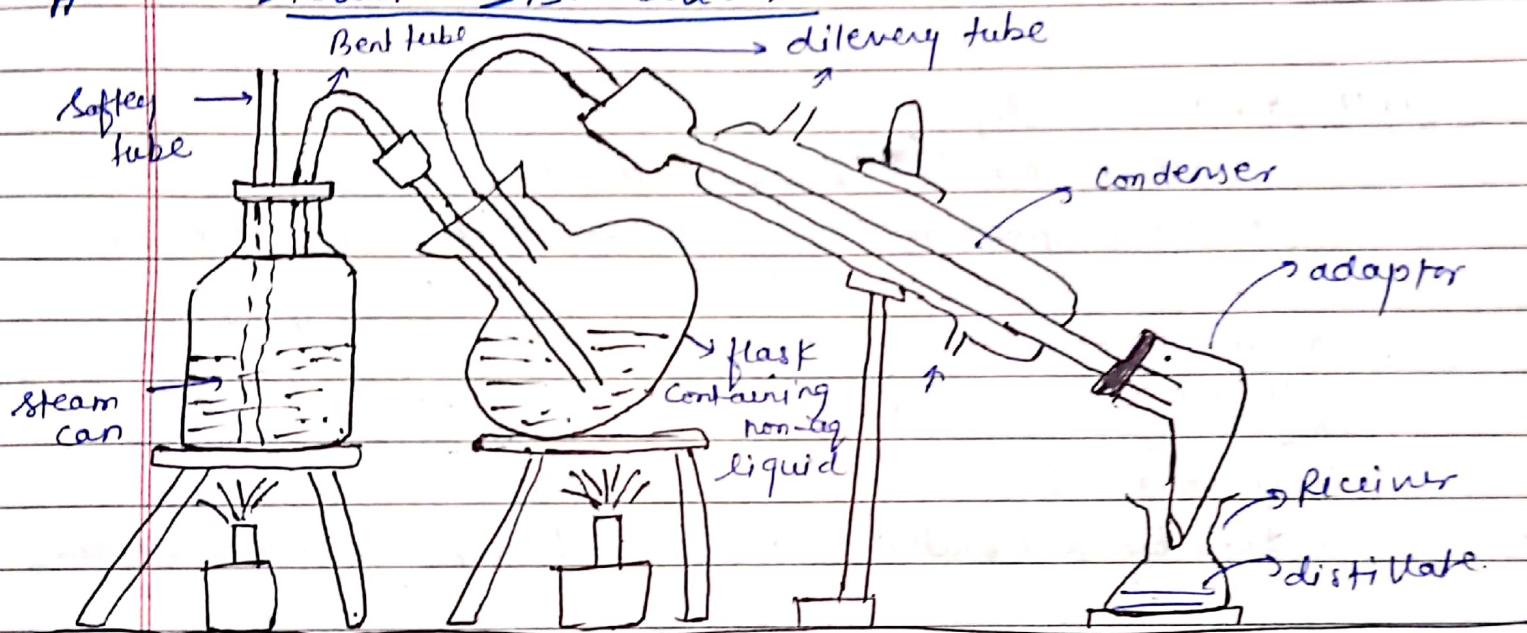
"The butterfly sleeps not months but moments, and has time enough." Rabindranath Tagore

Applications

- Purification of chemicals (dibutyl phthalate & dimethyl phthalate)
- more frequently used in refining of fixed oils.
- Vit A is separated from fish liver oil.
- Vit E is conc. by this process from fish liver oils.
- free fatty acids distilled at 100°C .
- steroids obtained at 100°C & 200°C
- triglycerides " from 200°C onwards.

#

Steam Distillation



→ It is method of distillation carried with the aid of steam & is used for the separation of ↑ boiling substances from non-volatile impurities.

→ It is most common ex. of differential distillation.

Principle

- immiscible liquid mix → boils (when sum of their VP = atm. press.)
- water + turpentine, mix. boils $<$ BP of pure H_2O , though turpentine boils at \gg temp than that of water.

BP of turpentine = 160°C , • water + turp. → boils at 95.6°C

Thus ↑ boiling substances may be distilled at temp much below

$$\begin{aligned} \text{Sum} &= 101.31 \text{ kPa} \\ &\downarrow \end{aligned}$$

$$\left[\begin{array}{l} \text{VP of } \text{H}_2\text{O} = 86.2 \text{ kPa} \\ \text{VP of turp} = 15.06 \text{ kPa} \end{array} \right]$$

its BP, when water is used, normal atm. press.

- For volatile sub., mis. with H_2O , steam dist. has same principle as that in Fn.

"Faith is the bird that feels the light when the dawn is still dark." Rabindranath Tagore

- Const → a metallic steam can + cork with 2 holes
 one / other → a bent tube is passed.
 - Other end of bent tube
 ↓
 connected to a flask containing non-ag. liq (crude containing volatile oil)
 thru rubber bung, other hole of rubber bung → a delivery tube is inserted → connects flask to condenser receiving adapter
 - If steam comes out, means steam can is empty.
- Provisions for heat' steam can & flask.

- Workn →
 - non-ag. liq → flask + small quant. of H₂O
 - steam-can → filled with H₂O.
 - steam generator & flask → heated simultaneously

steam carries ← mix gets heated ← uniform flow of steam passes thru boiling mix.
 volatile oil & passes into condenser

↓
 cooled by cold water → condensed immis. liq collected in receiver

- distillatn continues until non-ag. liq has been distilled.
- In receiver, the org. liq form 2 separate layers, which is separated in a separating flask.

- App →
 - To separate immis. liq. (toluene & water)
 - Extract most volatile oils (cloue, arise (eucalyptus))
 - Purificatn of liq with TBP (essential oil of almond)
 - Camphor is distilled by this method.
 - Aromatic water prepared.

- Adv → Vol. oils can be separated at ↓ temp, w/o decomposit' or loss of aroma. ↓ volatility sub. can be distilled (mw↑)
- Disad → Not suitable when immiscible liq & water react with each other.