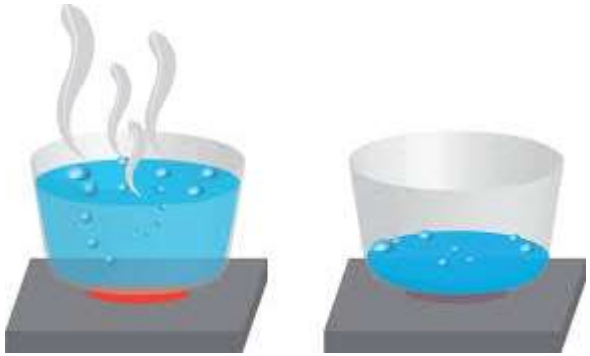
SS INSTITUTE OF PHARMACY

UNIT–2

EVAPORATION



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Contents

- Objectives
- Applications
- Difference between evaporation and other heat processes
- Factors influencing evaporation
- Various equipments of evaporation: Principle, working, construction, uses, merits and demerits

Concept of evaporation

- It is the free escape of vapour, from the surface of liquid, below its boiling point.
- Vapour is not collected but is allowed to diffuse into the atmosphere.
- It is a slow process.
- It can take place at room temperature.

Definition of Evaporation

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

- Equipment used for evaporation are known as "Evaporators."
- Heat is supplied to the evaporator, which transmits it to the evaporating liquid and provide the latent heat of vaporization.
- Thus, no boiling occurs and the rate of vaporization depends on the diffusion of vapour through the boundary layers above the liquid.

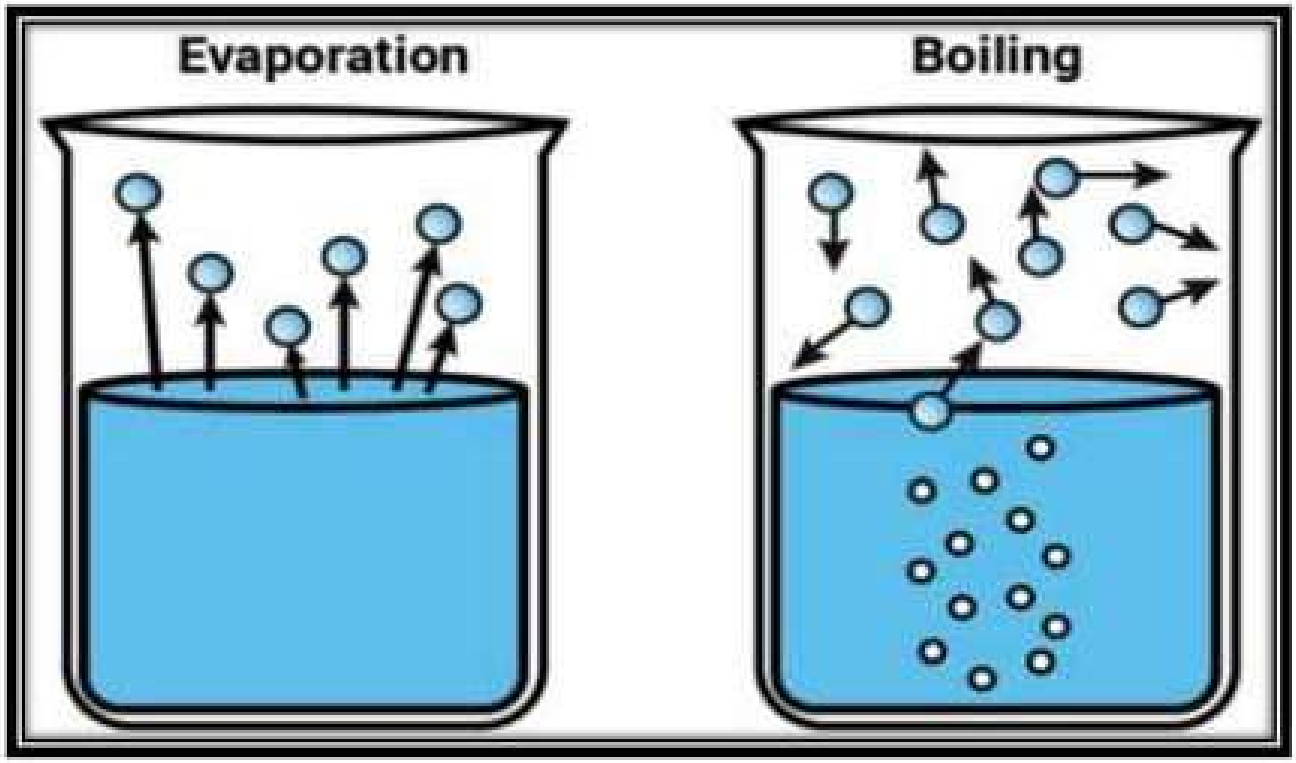
- Source of heat: Steam.
- It is a surface phenomena- i.e. mass transfer occurs from the surface of the liquid, so no boiling occurs resulting in the slow process.
- Solutions or suspensions can be subjected to the evaporation.
- **Conditions required for evaporation:**

i. Liquid should be volatile
ii. Solute must be nonvolatile
iii. As heat is supplied, constituents should be thermostable.

iv. In most operations, liquid is water with a low solid content.

v. At the end, concentrate will be so viscous that further the evaporation will be drastically reduced.

Difference between Evaporation and Boiling



Application of Evaporation

1. To get a concentrated product
2. To remove liquid from the solid product and to get the dry product.
3. For manufacturing of bulk drugs- used in pharmaceutical industries, chemical industries, etc.
4. For manufacturing biological products- E.g. insulin, bio-chemical products (penicillin), and plant products. Also for various blood products such as plasma and serum. Enzymes, hormones and antibiotics can also be prepared.

Difference between evaporation and other heat processes

Sr. No.	Evaporation	Other heat processes
1	Residue- Concentrated liquid.	Drying- residue is solid.
2	Evaporating liquid is only one component is most of the cases.	Distillation- It is a combination of 2 or more components.
3	Mixture of vapour is not separated.	Distillation- It is compulsory to separate each component.
4	Purpose- to get concentrated product not crystals.	Crystallization- purpose is to get crystals.

Factors affecting Evaporation

$$M = \frac{KS}{P}(b - b')$$

Where,

M = Rate of Evaporation (m^3/s)

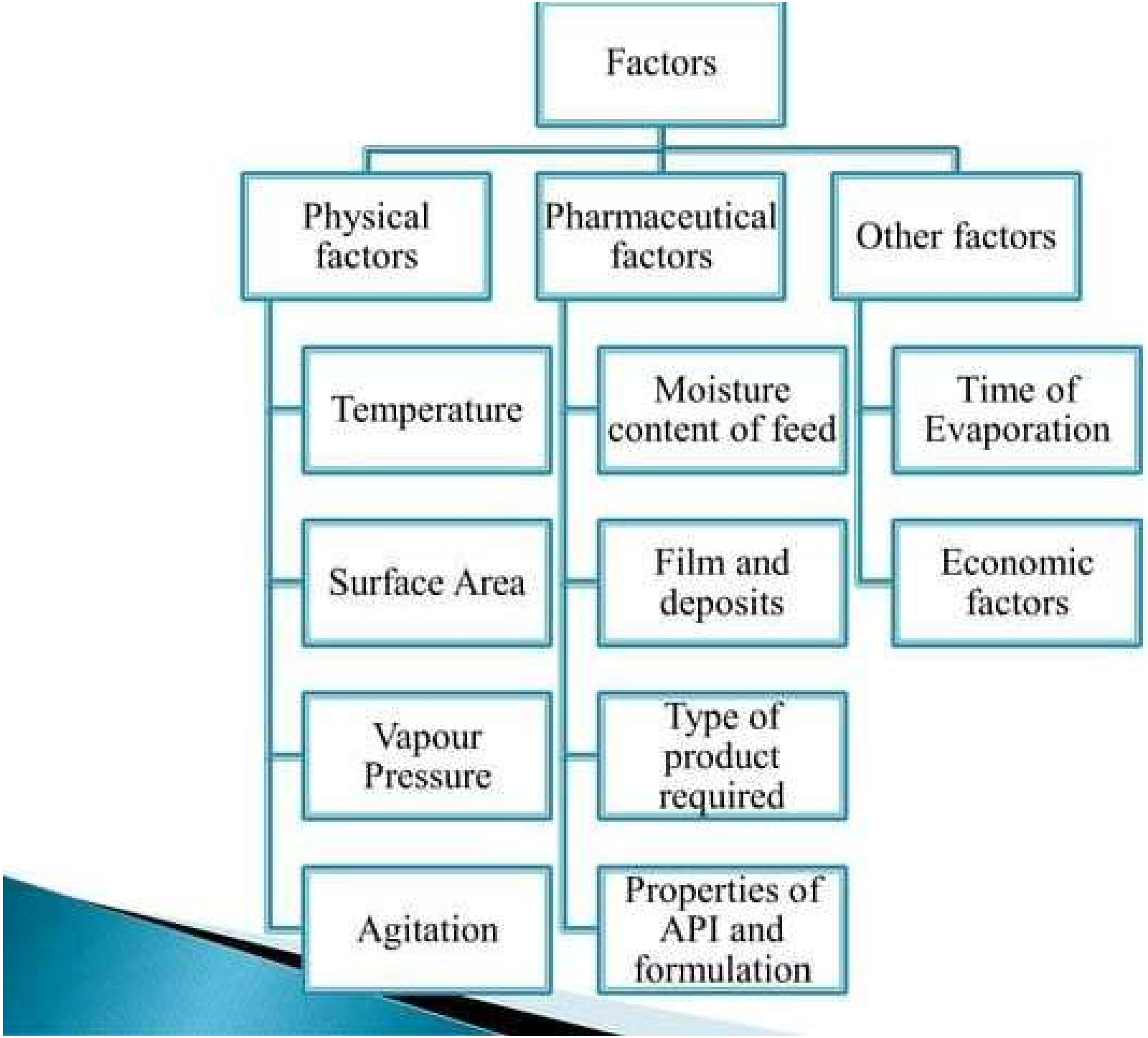
S = Surface area of liquid exposed to atmosphere (m^2)

P = Atmospheric pressure (kPa)

b = Max vapor pressure at temperature of Air (kPa)

b'= Pressure due to vapor of Liquid, actually present in air (kPa)

K = Constant (m/s)


Factors
Physical factors
Pharmaceutical factors
Other factors
Temperature
Surface Area
Vapour Pressure
Agitation
Moisture content of feed
Film and deposits
Type of product required
Properties of API and formulation
Time of Evaporation
Economic factors

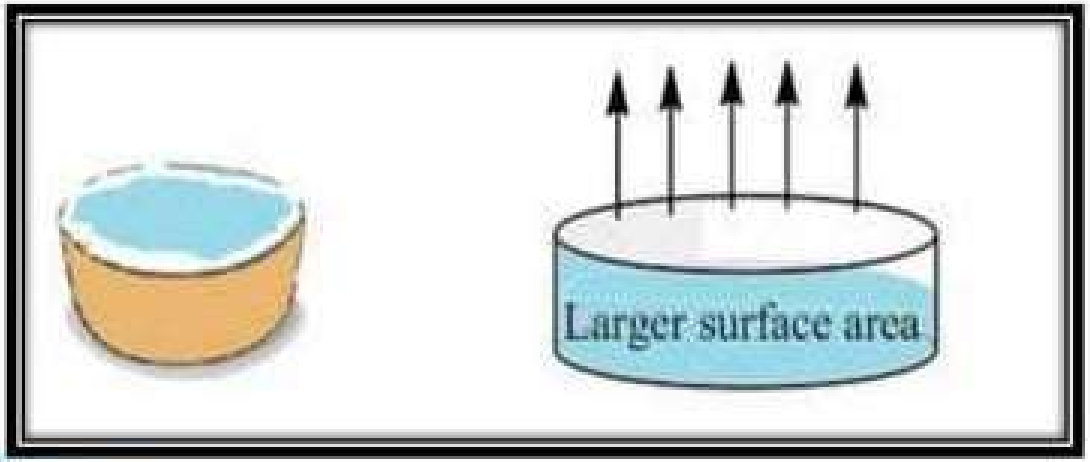
Temperature

- Higher the temperature, higher will be the value of b and thus greater will be the evaporation.
- The rate of evaporation is directly proportional to the temperature.
- At a given temperature, some molecules possess higher KE than average, while others have lower KE.
- Fast moving molecules escape from the surface of the liquid into vapour while slow ones remain behind.

- Increasing the temperature increases KE and escape of more molecules from the surface.
- Higher the temperature higher will be the evaporation.
- Evaporation is maximum at the boiling point of liquid. Below B.P, evaporation takes place only at surface. At B.P, vapours are formed throughout the surface of the liquid.
- Normally, glycosides and alkaloids decompose at high temperatures and hormones, enzymes and antibiotics are more heat sensitive thus, they require specialized techniques to prevent decomposition during evaporation
- Ex. Malt extract can be prepared to avoid loss of enzymes, antibiotics can be concentrated by freeze drying.

Surface Area

- The surface area of liquid exposed to evaporation is directly proportional to the rate of evaporation.
- Greater the surface area exposed to evaporation higher will the rate of evaporation.
- Ex. Film evaporation.



Vapour Pressure

- Rate of evaporation is directly proportional to the V.P.
- Lower the *p* value, greater is the evaporation.
- Lower the external pressure, lower is the B.P., greater is the evaporation. It is achieved by applying vacuum.
- Nature of liquid is also very important.
- Liquids with low B.P. evaporate quickly because of high V.P. at lower temperatures.
- If the outer atmosphere is dry, the value of *b*' will be low and hence greater is the evaporation.

- If the vapour of the liquid is removed as soon as it is formed, the space above the liquid does not become saturated with vapour and hence evaporation proceeds faster.

Agitation

- Agitation: More the agitation, better is heat and mass transfer.
- During evaporation liquid form a scum or layer on the top which lowers the rate of evaporation.
- During agitation or stirring the scum will break and rate of evaporation increases.
- It also prevents decomposition of the preparation at the bottom.
- As liquor gets concentrated due to evaporation, leads to elevation in boiling point.

- Due to higher boiling point more damage happens to thermolabile material.
- Concentrated liquor have higher viscosity and thicker boundary layers.
- The above problem can be minimised by agitation.

Properties of API and Formulation

- Thermostable preparations can be evaporated at high temperatures.
- Thermolabile preparations will have to be evaporated at low temperature.
- For antibiotic possible method is freeze drying.
- Some drugs decompose more readily in the presence of moisture at higher temperature.

Type of product required

- Type of product required will often decide which method and apparatus should be used.
- Evaporating pans or stills will produce liquid or dry product.
- Film evaporators will yield only liquid products.
- Vacuum apparatus can be used to obtain dry porous product. Ex. granular extract of cascara

Moisture content of feed

- Some drugs undergo hydrolysis in presence of moisture at higher temperatures.
- Evaporation may be carried out at low controlled temperature till a concentrated product is obtained and then final drying at high temperature when little moisture remains.
- Ex. Belladonna dry extract

Film and deposits

- When vegetable extracts are concentrated in pans it forms film on surface of the liquid and deposit at the bottom.
- The film reduce evaporating surface and deposit hinders the transfer of heat.
- Both these problems may be minimised by effective stirring. Ex. Agitated Evaporators.

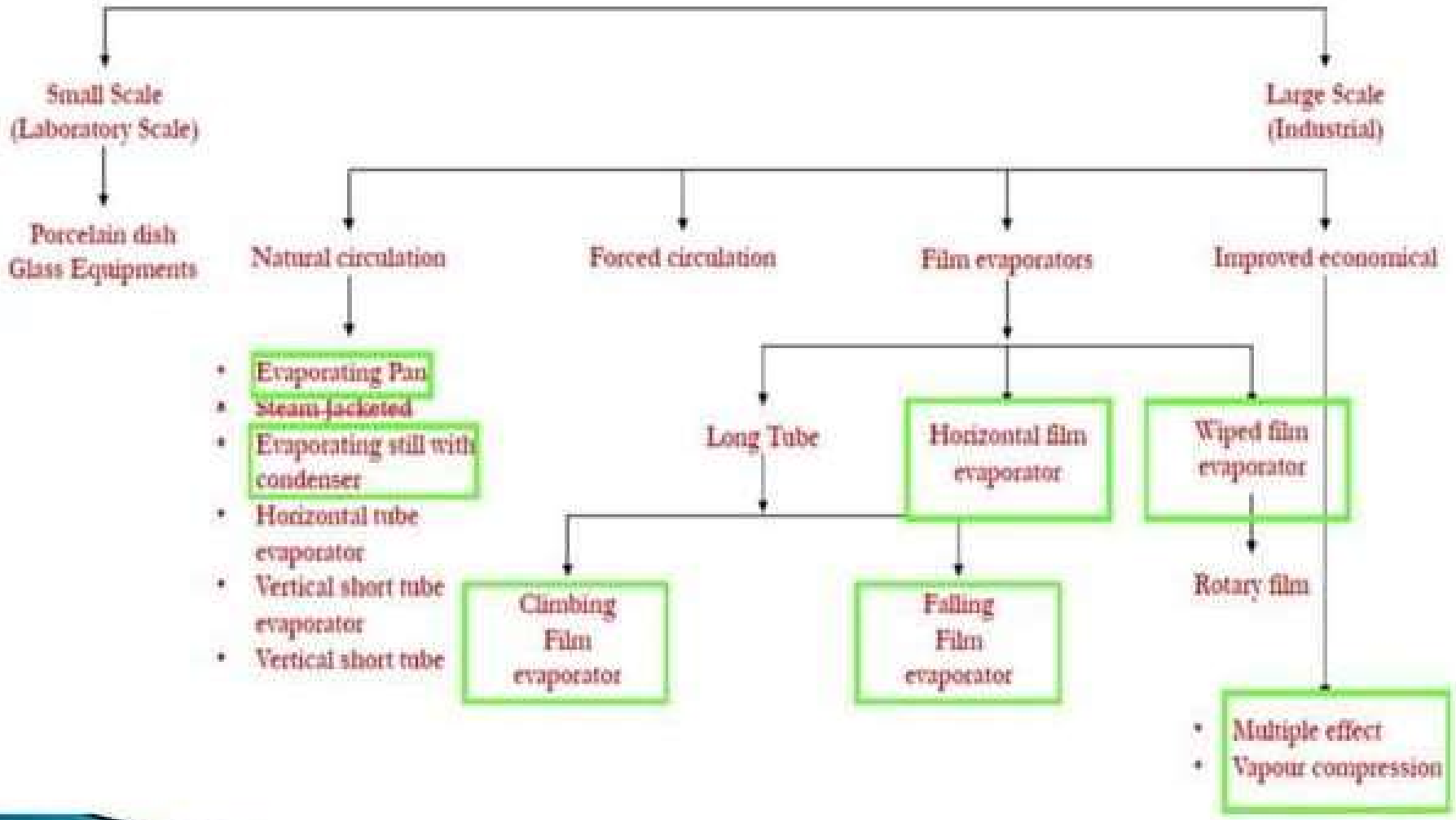
Time of evaporation

- If the time of exposure is long, greater will be the evaporation, provided the constituents are thermostable.
- Exposure of drug to a high temperature for a short period of time may be less destructive of API than a lower temperature with long exposure periods.

Economic factors

- Nature of liquid: Liquids with low boiling points evaporate quickly because of high vapour pressures at low temperatures e.g. Volatile liquids.
- Exposure to high temperature for a short period of time may be less destructive than a lower temperature with exposure for a longer period.
- Film evaporators use high temperature for a short period whereas an evaporating pan may involve prolonged heating.
- Economies of labor, fuel, floor space and material are of primary considerations. Recovery of solvent and utilization of waste heat also need to be considered.

Types of evaporators



Components of an Evaporator

- Indirect contact heat exchanger (steam) - Supplies sensible and latent heat
- Vacuum pump-Temperature between steam & product increases
- Vapour- liquid separator - separate vapour (undesirable) from product (desirable)
- Condenser - to condense vapour and remove it from system
- Pre-heater (optional) - increases efficiency of operation

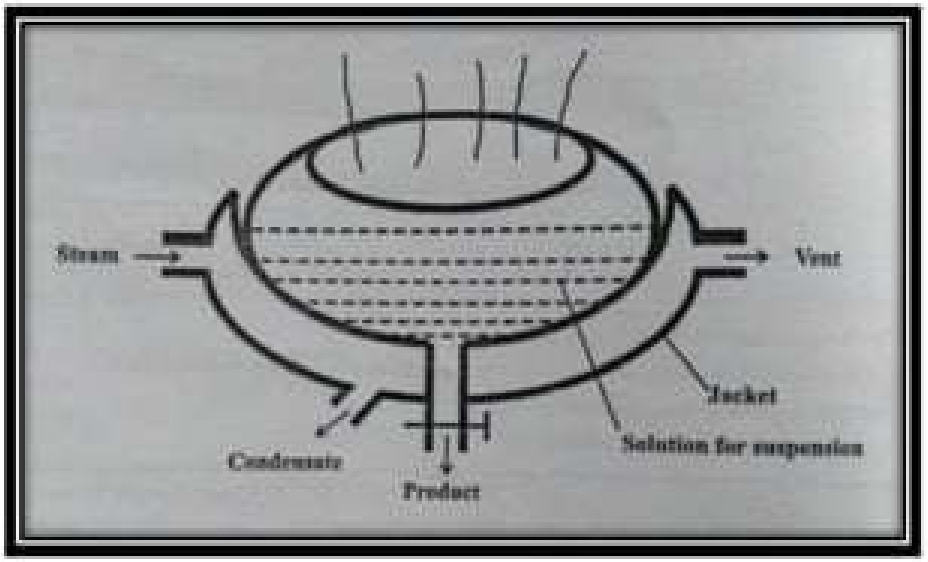
Natural Circulation Evaporators

- Evaporators of this category are those in which the movement of the liquid results from convection currents sets up by the heating process.
- Convection currents: The process in which heat moves through a gas or liquid as the hotter parts rises and the cooler part sinks

Steam Jacketed Kettle or Evaporating Pan

Principle

- Steam is supplied to jacketed kettle in which liquid extract is placed.
- Steam gives out heat to the kettle.
- Heat is transferred to aqueous extract by conduction and convection
- As temperature raises escaping tendency of the solvent molecules into vapour increases.
- Stirring also increases vaporization.



Construction

- Hemispherical structure consisting of inner pan- Kettle, enveloped with outer pan- Jacket
- 2 pans are joined to enclose a space from which the steam is passed.

- Great variety of materials for construction: E.g. Cast iron (tinned/enameled to prevent rusting), stainless steel, copper, tinned copper (for acidic materials), aluminum.
- Iron is used for construction of jacket, because it has minimum conductivity.
- Kettle may be made of single metal sheet or several sheets joined by welding/brazing
- Outlet (Vent) – for non condensed gases
- Outlet for product discharge at the bottom/ tilting type for smaller capacity (<90lts)

Working

- Aqueous extract- kettle
- Steam is supplied through the inlet.
- Steam gives heat to contents and condensate leaves through the outlet.
- For smaller volume of contents they should be stirred manually and for larger volumes they should be stirred mechanically.
- R.O.E is fast in initial stages but decreases gradually because liquid becomes concentrated.

- Heat transfer – Conduction and convection.
- Rate of heat transfer varies from 50-300 Btu/(sq. ft)(°F)(hr) (Btu= British Thermal Units)
- Depends on viscosity of liquid, amt of agitation and material type.
- Rate of evaporation is fast initially & decreases gradually as liquid gets concentrated.
- Agitation is necessary.
- Good ventilation required to remove vapor.
- Fans fitted over pan removes vapor & prevents condensation in room.

Uses

- Concentrating aqueous & thermo stable liquors. E.g. Liquorice extract

Advantages

- Both small and large scale operations.
- Simple in construction and easy to operate, clean and maintain.
- Cost of installation and maintenance is low.
- Wide variety of material can be used for construction.
- Stirring of contents and removal of product is easy.

Disadvantages

- Heat transfer areas normally are quite small due to vessel shapes, and heat transfer coefficients tend to be low under natural convection conditions
- Heat economy is less. Thus, cost per unit material production is more.
- Not suitable for heat sensitive materials due to long time of exposure.
- Heating area decreases as product gets more concentrated.
- In open type kettle, vapor passes into atm, which can lead to saturation of atm, slowing evaporation & discomfort.

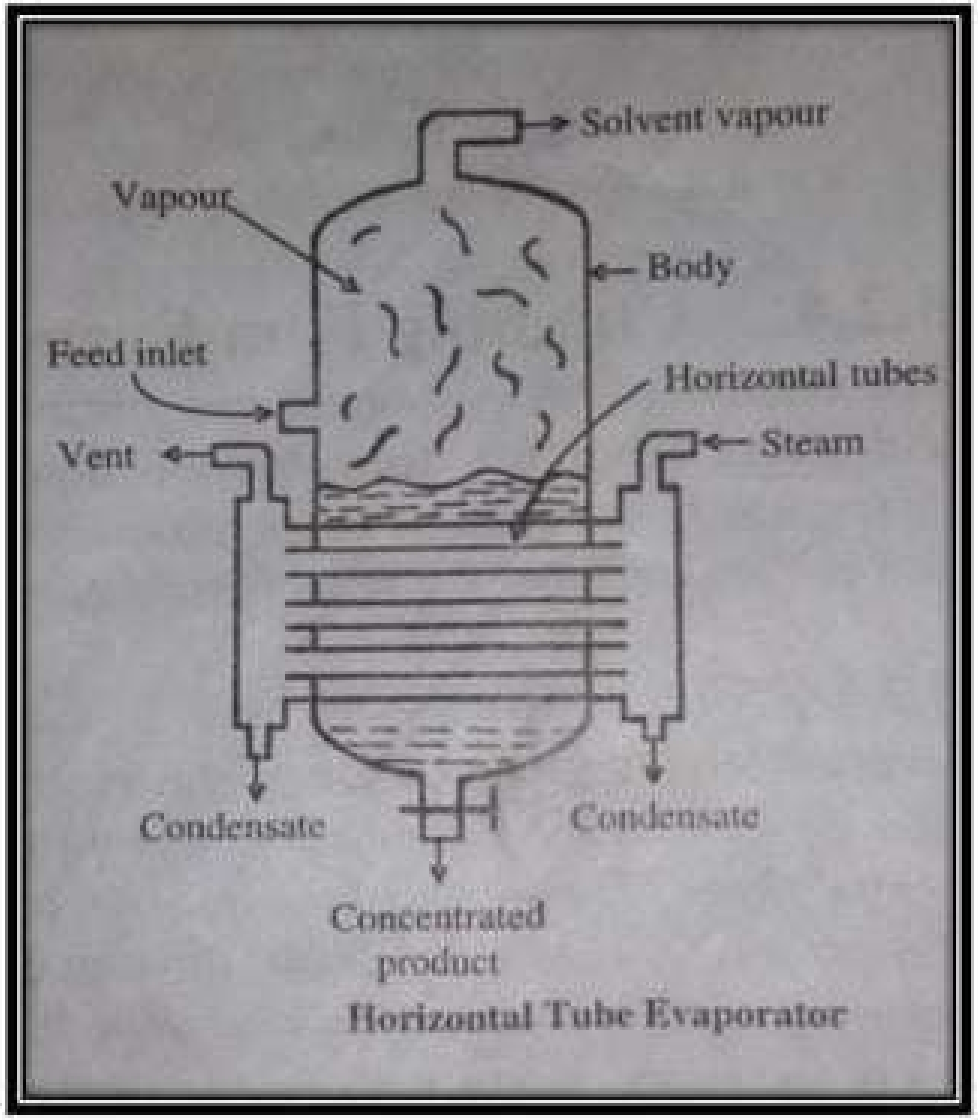
Horizontal (Short tube) Evaporator

Principle

- It is based on the principle of natural circulation.
- Steam is passed through horizontal tube of evaporator which is immersed in pool of liquid to be evaporated.
- Heat transfer takes place through the tubes and liquid outside the tube gets heated.
- The solvent evaporates and escape from top of evaporator.
- The concentrated liquid collected from bottom

Construction

- It consist of large cylindrical body with conical or dome shaped top & bottom .
- Made-up of cast iron or plate steel Avg diameter : 1.8 to 2.4 meter, Avg Height : 2.4 to 3.6 mtr.



Horizontal Tube Evaporator

- Lower part consist of steam compartment (Half of the body of cylindrical body) with inlet and vent outlet for non condensed gases on the other end.
- Condensate outlet is present at the bottom of steam compartment.
- Steam compartment consist of 6-8 SS horizontal tubes are placed.
- Width of the steam compartment is half of the diameter of the body.

Working

- Feed is introduced into evaporator untill the steam compartment is satisfactory immersed.
- Steam is introduced into the steam compartment.
- Heat transfer takes place through the tubes and conduct it to the liquid compartment due to temperature gradient.
- Steam condensate passes through corresponding outlet.
- Feed absorbs heat and solvent get evaporated.
- Vapour than escapes through the outlet placed at the top.
- This process is continued until thick liquid is form which can be collected from the bottom outlet.

Advantages

- Cost of operation is low.
- Efficient utilization of heat.
- Easy to construct and maintain.
- Stirring and removal of product is easy.

Disadvantages

- Difficult to clean.
- Not suitable for thermolabile material.
- Cost of installation and maintenance is high.

Application

- Suitable for non viscous liquid that do not deposit scale or crystal on evaporation (example: Cascura extract)

Climbing Film Evaporator (Rising Film Evaporator)

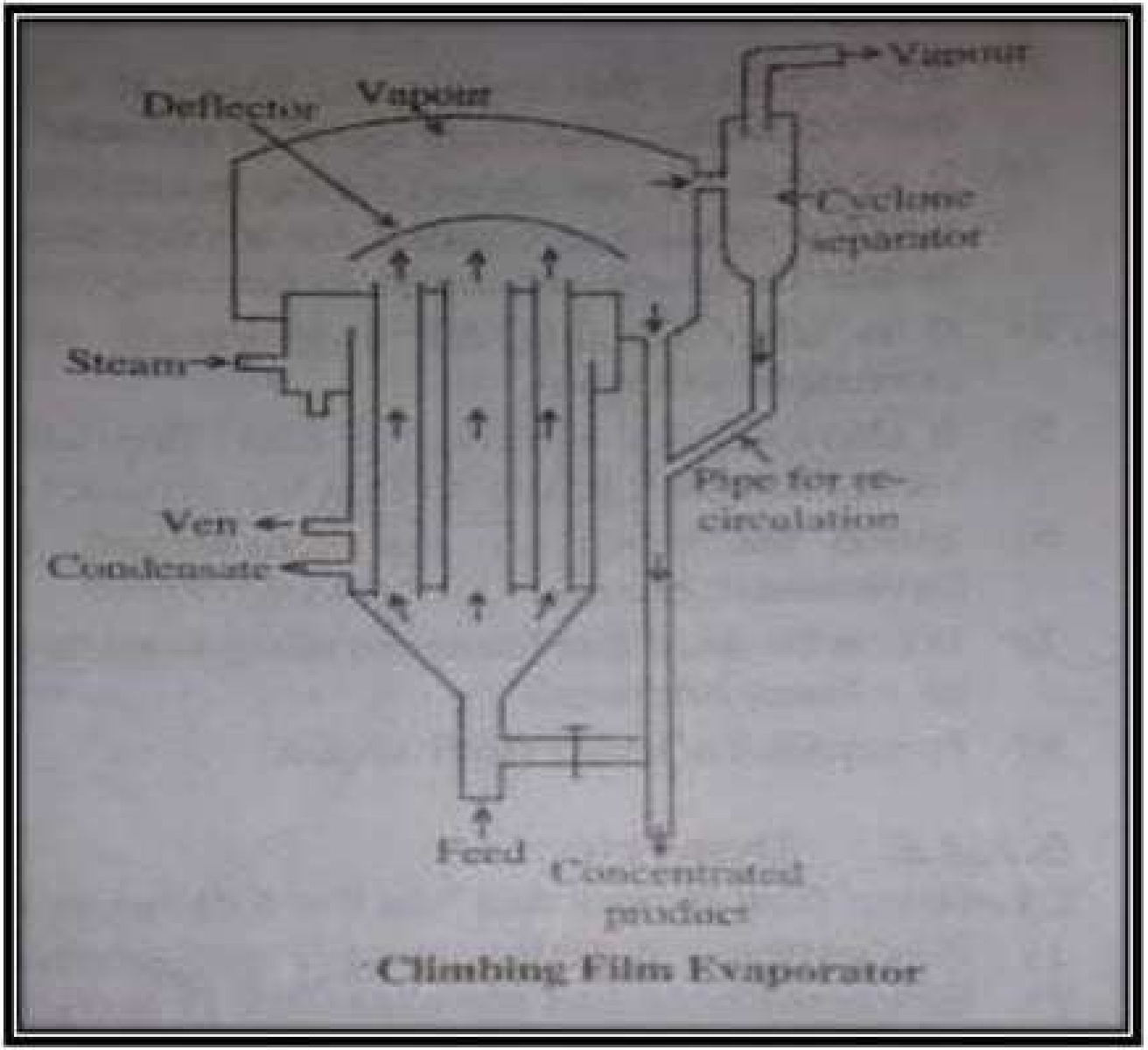
Principle- Natural circulation with climbing film

- Tubes are heated externally by steam.
- The preheated feed enters the bottom and flows up through the heated tubes.
- Liquid gets heated rapidly due to enhanced overall coefficient of the preheated feed.
- Liquid near the walls become vapour and forms small bubbles.

- These tend to fuse to larger bubbles, that travel up in tubes with the entrapped slug.
- The liquid films are blown up from the top of tubes and strikes the deflector kept above.
- This throws the liquid concentrate down into the lower part from where it is withdrawn.
- The liquid films travel at a velocity of 6-7 m/s while vapour enters the separator at 27 m/s.

Construction

- Heating unit consists of steam jacketed tubes.
- The tubes are held between two plates.
- Length to diameter ratio is greater than 150.
- The tubes are 5 mm in diameter and about 7 m in length.
- Deflector is placed at the top of the vapor head.
- Inlets are provided for steam and feed.
- Outlets are provided for vapor, concentrated product, non condensed gases and condensate.



Climbing Film Evaporator

Working

- Preheated liquid feed is introduced from the bottom of the unit.
- Steam enters into the spaces outside the tubes through inlet.
- Heat is transferred to the liquor through the walls of tubes.
- Liquid becomes vapour and form smaller bubbles, which tend to fuse to form larger bubbles.
- Thereby, the bubbles trap a part of the liquid on its way up in the tubes.
- As more vapour is formed, the slug of the liquid is blown up in the tube and thus spread as a film over the walls.

- This film continues to vaporize rapidly.
- Finally, the mixture of the liquid concentrate and vapour eject at a high velocity from the top of tubes.
- Then entrainment separator not only prevents entrainment, but also act as a foam beaker.
- Vapour leaves from top while concentrate is collected from bottom.

Applications

- Perfect for pharmaceutical use.
- Concentration of solutions such as insulin, liver extracts and vitamins.
- For clear, foamy and corrosive liquids.

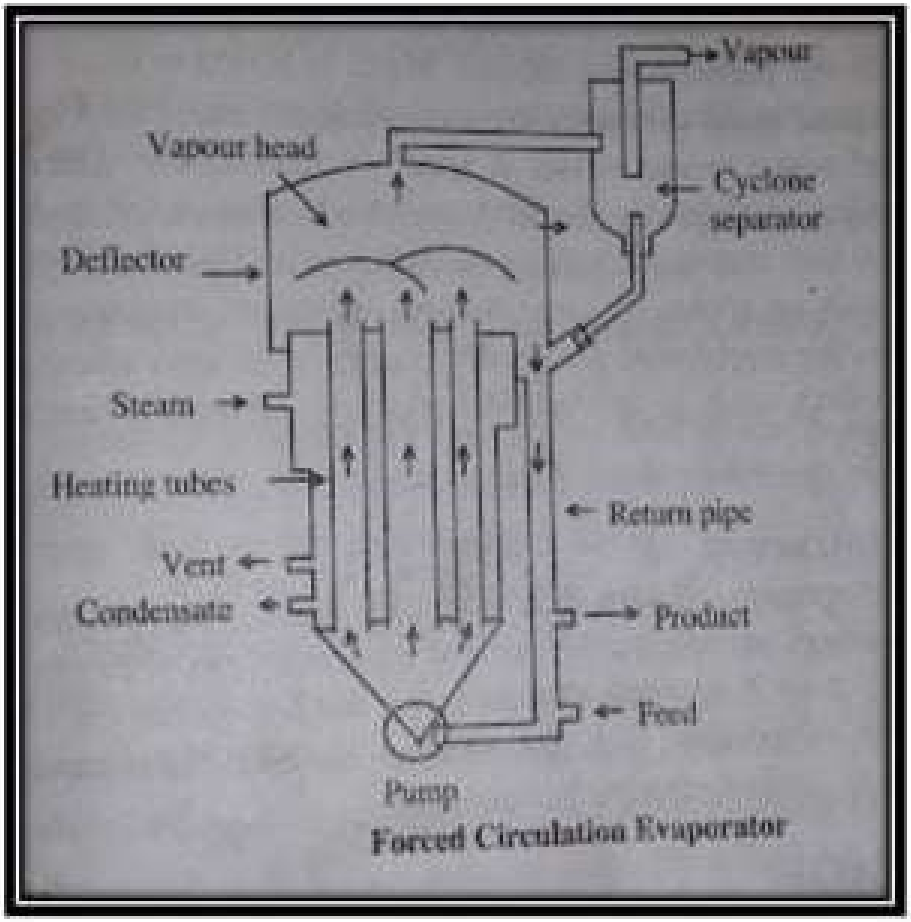
Advantages	Disadvantages
Large area for heat transfer is provided.	Expensive & complex construction
As liquid flows at a high velocity, resistance for heat transfer at the boundary layers is reduced.	Difficult to clean and maintain
The time of contact between the liquor and heating surface is short. Thus it is suitable for heat sensitive materials	Large head space is required.
Suitable for foam forming liquids as foam can be broken in an entrainment Deflector.	Not advisable for scaling liquids and viscous liquids
Requires low floor space.	

Forced Circulation Evaporator

- Liquid is circulated at high velocity through the heat exchanger tubes to enhance the heat transfer rate and inhibit particle deposition.
- Any evaporator that uses pump to ensure higher circulation velocity is called a forced circulation evaporator.
- A centrifugal pump forces liquid through the tubes at an entering velocity of 2 to 5.5m/s.
- Heat transfer coefficients increases enormously and it also prevents the scale formation on heating surfaces.

Principle

- Liquid is circulated at high pressures - Boiling point is elevated.
- Forced circulation creates some form of agitation.
- When the liquid enters the vapour head, pressure falls suddenly.
- This leads to the flashing of super heated liquor which leads to evaporation.



Forced Circulation Evaporator

Construction

- The tubes are held between two tube sheets and measures 0.1 m inside diameter and 2.5 metres long and they are longer and narrower than vertical evaporator.
- The main components of a forced circulation evaporator are: a tubular shell, tube heat exchanger (either horizontal or vertical), flash chamber (separator) mounted above the heat exchanger, and circulating pump.

Working

- Steam is introduced into calendria (consists of number of tubes).
- Pump sends liquid to the tubes with a positive velocity.
- As liquid moves up through the tubes, it gets heated and begins to boil.
- The vapour and liquid mixture rushes out of the tubes at a high velocity.
- This mixture strikes the deflector, which throws the liquid downward.
- This result in an effective separation of liquid and vapour.
- The vapour enters the cyclone separator and leaves the equipment. The concentrated liquid returns to the pump for further evaporation.
- Finally concentrated product is collected.

Advantages

- Heat transfer coefficient is high.
- Salting, scaling and fouling is not possible due to forced circulation.
- Suitable for viscous preparations.
- Moderately heat sensitive liquid can be concentrated.

Disadvantages

- In FC evaporator, the hold-up of liquid is high.
- The equipment is expensive, because power (pump) is required fro circulating the liquid.

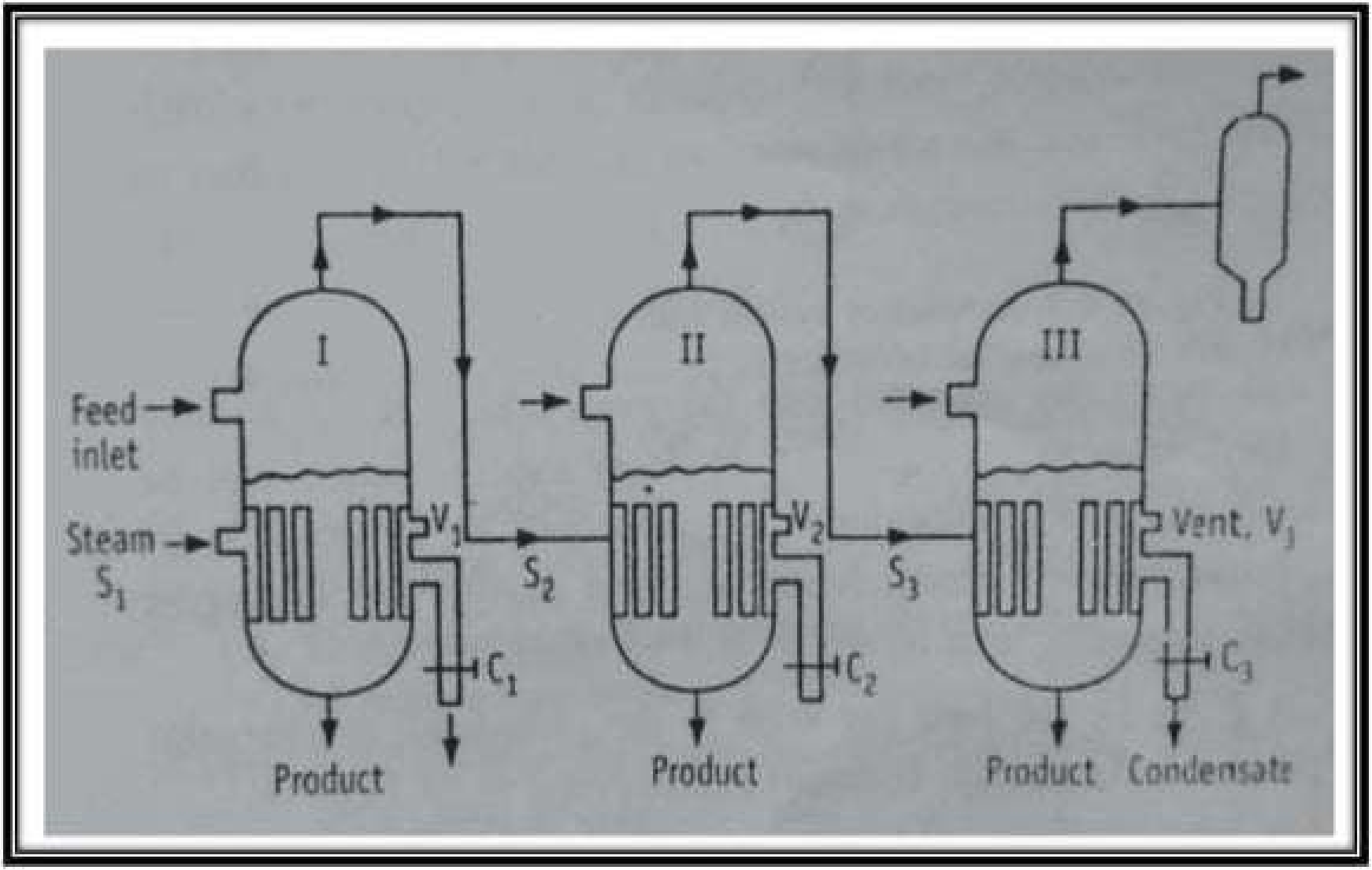
Uses

- Reduced pressure - thermolabile substances.
- Conc. of insulin and liver extracts.
- Suitable for crystallizing operations.
- Viscous liquids and Suspended solids.
- Concentration of caustic and brines solutions and evaporation of corrosive solution

Multiple Effect Evaporator

- Simple evaporators that were studied earlier are connected in different ways to achieve large scale evaporation as well as greater economy.
- They are not widely used in pharmaceutical industries, but the principles are of interest and they should be understood.
- To avoid the wastage, two evaporators are connected together with the piping arrangement so that the vapour from the calendria of the first effect is used to heat the calendria of the second effect .

- This means that the calendria of the second effect is used as a condenser for the first time. So that the latent heat of vaporization is used to evaporate more quantity of the liquid instead of its going as waste.
- The vapor from the second effect is then taken to a condenser and converted in to the liquids.
- In general not more than three or four effects are combined together to have economical and efficient evaporation of liquids.


I
II
III
Feed
inlet
Steam
S_1
V_1
V_2
Vent, V_3
S_2
S_3
C_1
C_2
C_3
Product
Product
Product
Condensate

Working

- At start, the equipment is at room temperature and the atmospheric pressure.
- The liquid feed is introduced to all the three evaporators up to the level of the upper tube sheets.
- The vent valves V_1, V_2, V_3 are kept open and all other valves are closed.
- Now a high vacuum is created in the liquids chambers of evaporators.
- The steam valves S_1 and condensate valves are opened. Steam is supplied.
- Steam first replaces cold air in the steam space of 1st evaporator.

- When all the cold air is removed the valve V_1 is closed.
- The supply of steam is continued until the desired pressure P_0 is created in the steam space of 1st evaporator.
- At this pressure the temperature of the steam is t_0.
- Steam gives its temperature to the liquids feed in the 1st evaporators and gets condensed.
- Condensate is removed through the valve C_1.
- Due to the heat transfer the liquids temperature increases and reaches the boiling point.
- During this process vapour will be generated from the liquid feed.
- Formed vapour displaces air in the upper part of 1st evaporator.
- Moreover the vapour also displaces the air in the steam space of the 2nd evaporator.

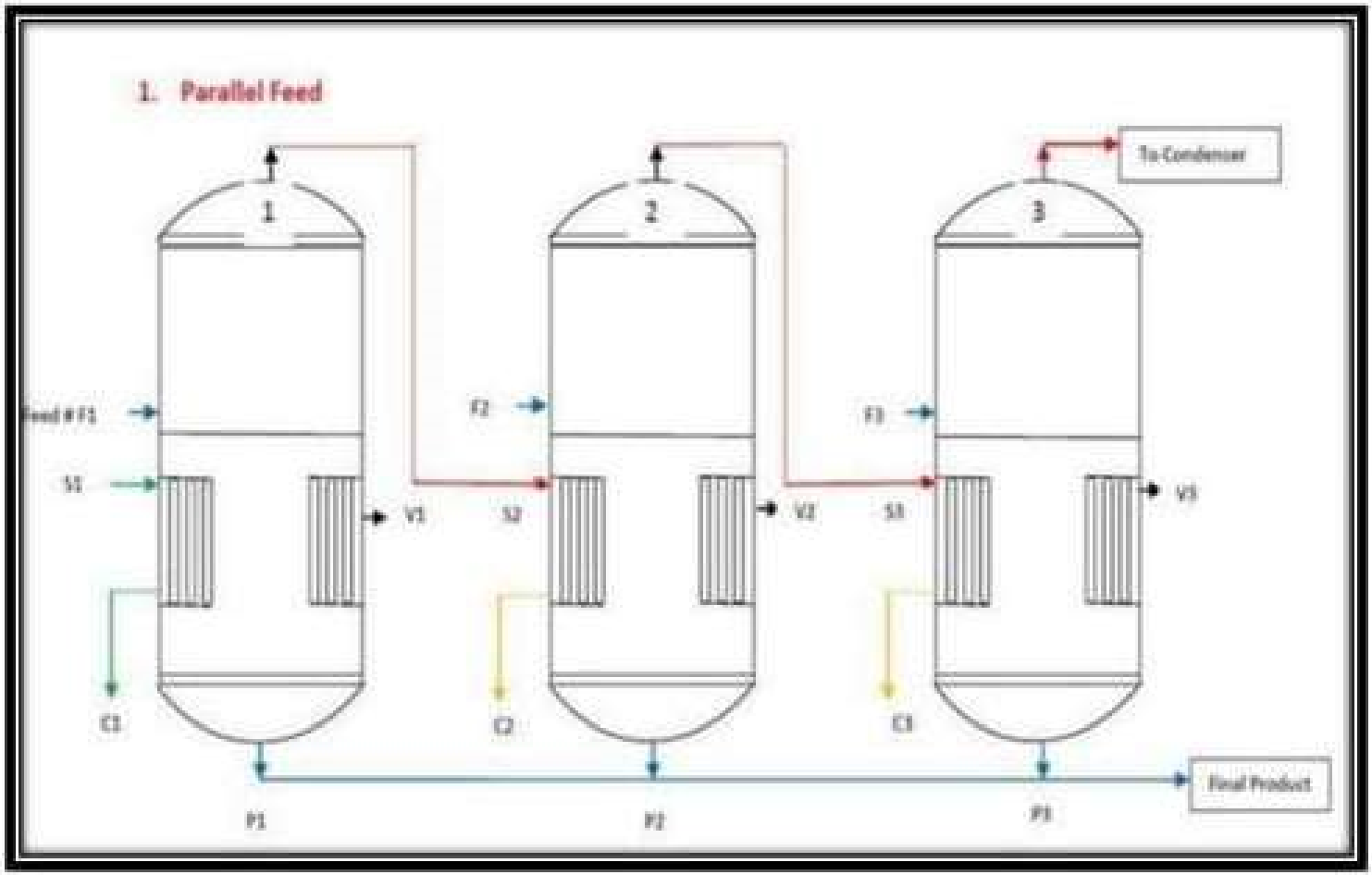
- After complete displacement of air by vapour in the steam compartment of 2nd evaporator, valve V_2 is closed.
- The vapour of 1st evaporator transmit its heat to its liquid of the 2nd evaporator and gets condensed, condensate is removed through the valve C_2.
- These steps continue in the 3rd , 4th evaporator also.

- The pressure in the vapour space of the 1st evaporator gradually increases to P_1 by increasing temperature to T_1 which is the boiling point of the liquid in the first evaporator and decreasing the temperature difference. $(t_0 - t_1)$
- Similar changes take place in 2nd and 3rd evaporator till a steady state is reached with the liquid boiling in all three bodies.
- As the boiling proceeds, liquid level in 1st evaporator comes down. Feed is introduced into the feed valve to maintain a constant level.
- F_2 and F_3 inlets are used to provide feed to 2nd and 3rd evaporator to maintain the desired levels.
- The process is continued till the product in all three effects reaches the desired viscosity. The products valves are opened to collect the thick liquid
- Continuous supply of steam, feed and continuous removal of product from all three evaporators – continuous process.

Methods of feed introduction

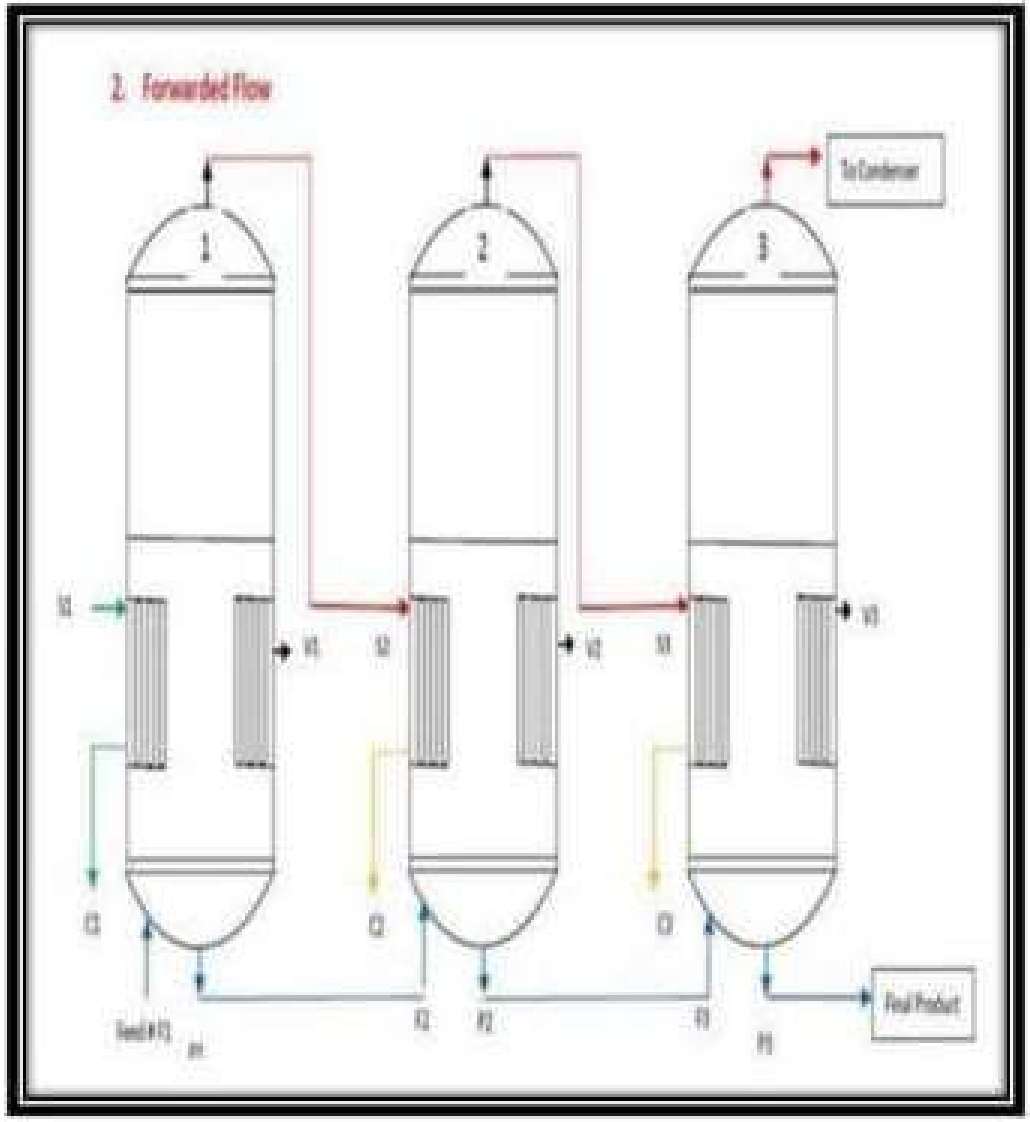
1. Parallel feed

- In this evaporator there is a continuous supply of feed continuous supply of steam and continuous withdrawal of liquid from all the three evaporators.
- Hence the evaporators works continuously with all the temperature and pressure in balance.


1. Parallel Feed
1
2
3
To Condenser
Feed # F1
F2
F3
S1
S2
S3
V1
V2
V3
C1
C2
C3
P1
P2
P3
Final Product

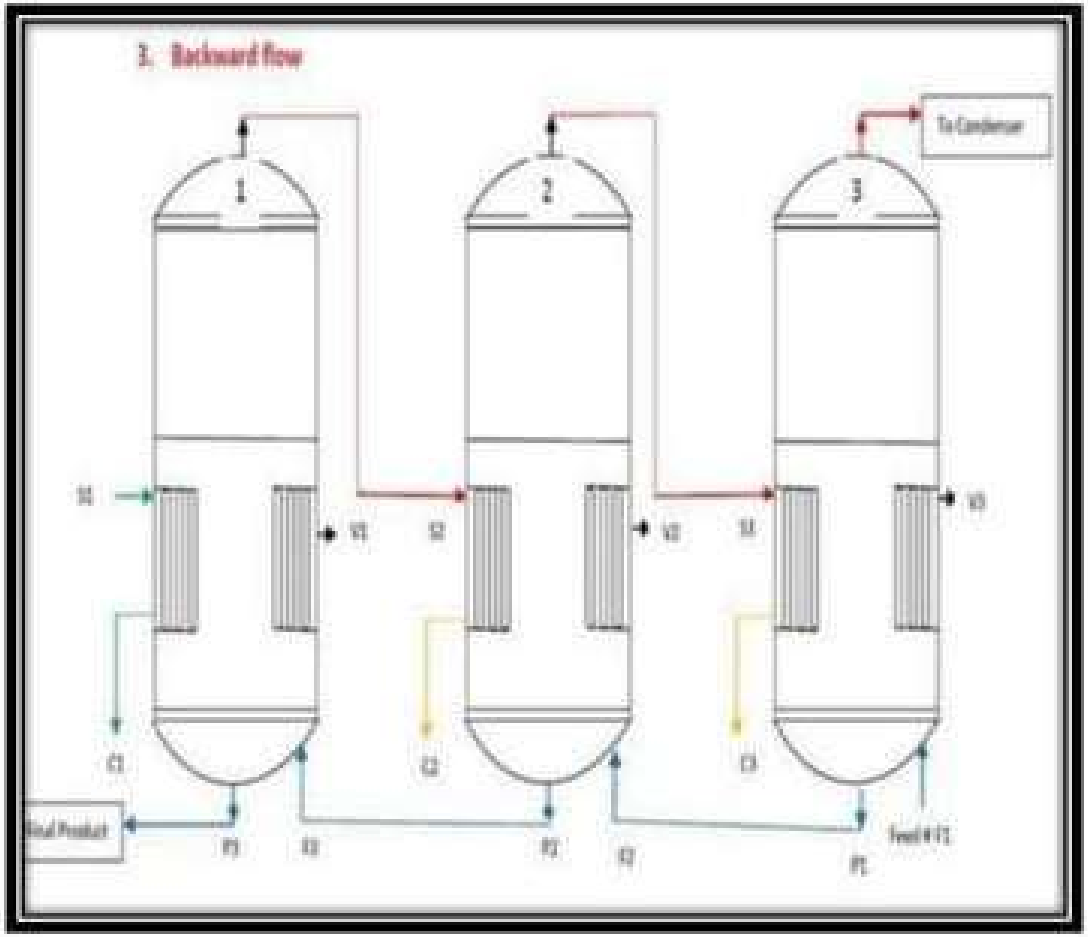
2. Forwarded feed flow

- In the forward feed method the mother liquor is introduced in to 1st then transferred to 2nd and then to 3rd , 4th.



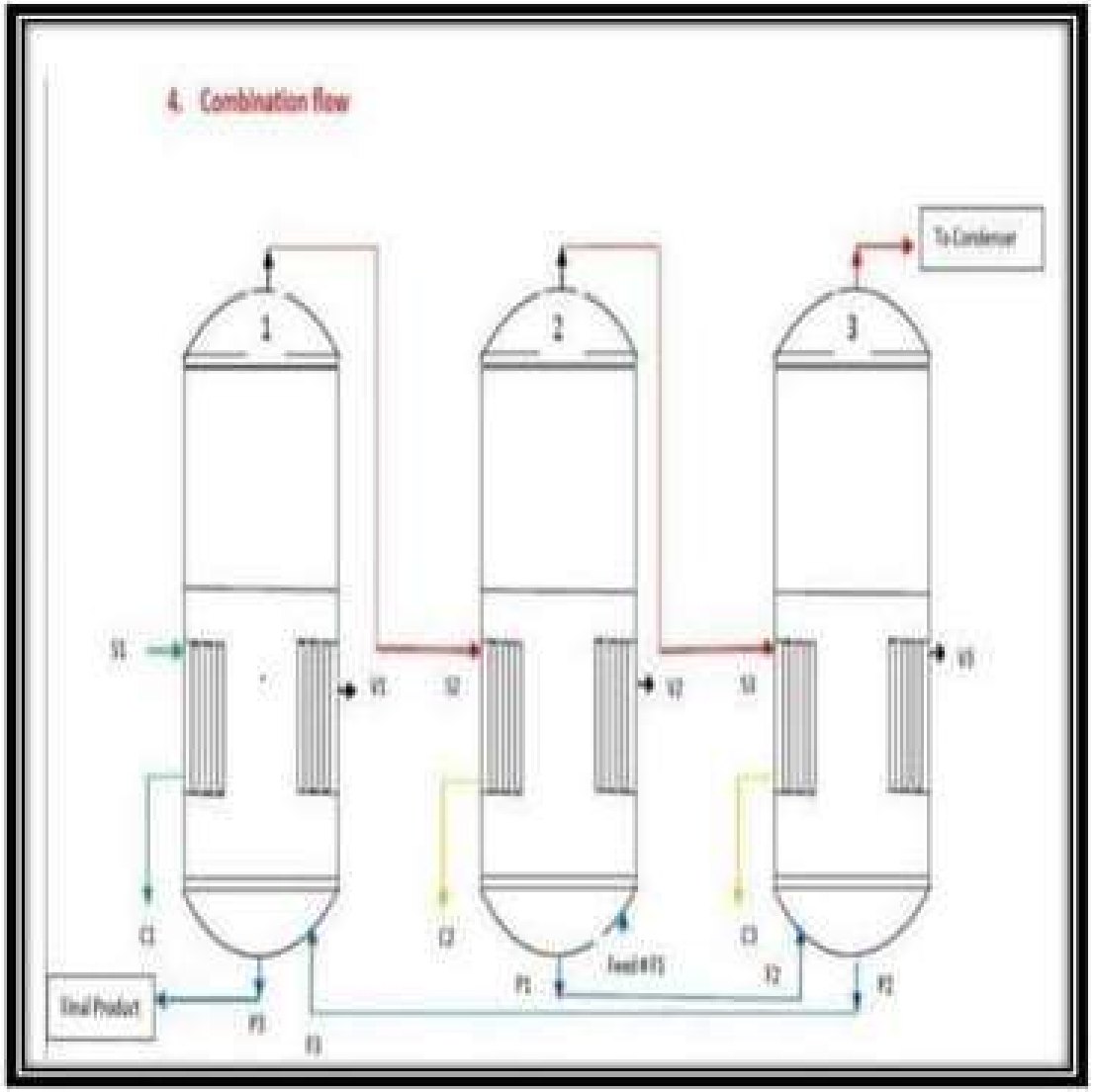
3. Backward feed method

- In this method, the mother liquor is introduced in to the 4^{th} evaporator, then transferred to 3^{rd}, 4^{th} then transferred to 1^{st}



4. Combination flow

- Mixed feed method (Combination flow) , the mother liquor is introduced in to 2^{nd} and 3^{rd} evaporator then transferred to 4^{th} evaporator and then transferred to 1^{st} evaporator.



Advantages

- Suitable for large scale and for continuous operation.
- Highly economical compared to single effect evaporator.
- About 5 evaporators can be attached.

Economy of Multiple Effect Evaporator

- Amount of vapors produced per unit steam input.
- It is calculated by condensing the following Assumptions:
- Feed is admitted at boiling point (therefore, it does not require any more heat to raise its temperature)
- Loss of heat is negligible.
- Hence , the supplied steam gets condensed to give heat of condensation.
- This heat will then be transferred completely to the liquid.

- The heat transferred now serves as latent heat of vaporization, i.e. liquid undergoes vapourization by receiving heat. Loss by any means is negligible.
- One pound of steam introduced into first effect will vaporize N pounds of water in a multiple evaporator with N effects.

- Economy of a single effect evaporator = Total mass of vapour/total mass of steam supplied
- In single effect of evaporator, steam produces vapour only once.
- Hence economy = N units of vapour produced/N units of steam supplied = 1
- In multiple effect of evaporator, one unit steam produces vapour many times, depending upon the number of evaporators connected.
- Hence, Economy = N units of vapour produced/1 units of steam supplied = N
- Therefore, economy of multiple effect evaporator is N times the economy of the single effect evaporator.

- However, such a great economy is approximately true as it depends on many factors such as

1. Temperature of the feed
2. Temperature range in the evaporator,
3. Ratio of weight of feed to product
4. Pressure difference


Thank you