INDUSTRIAL PHARMACY
(LECTURE 5)

Extraction

Lecture V

Definition: Extraction is the process in which a soluble constituent present either as a solid or a liquid is removed from a solid or from a liquid mixture by the use of a selective solvent.

Extraction includes:

1- Solid - liquid extraction (leaching):

the plant cellular materials handled may be:

- Cellular materials
- Coarse materials
- Intermediate materials.
- Fine materials.

2- Liquid - liquid extraction.

Factors affecting the rate of leaching:

1- Particle size:

- As the particle size decreases the surface area of the solid exposed to the solvent increases leading to increase in extraction rate.
- The particle size should be optimal as further decrease in the particle may lead to decrease channeling of the solvent through the solid bed thus decrease the extraction rate.

2- Solvent:

a- Solvent viscosity: By increasing the viscosity , the movement of the solvent through the bed of the solid become slow.

- Also the viscosity affects the diffusion of soluble solid in the liquid. Thus the rate of extraction is inversely proportional to the viscosity of the solvent.

b. The solvent should be selective: The solvent should be selective to dissolve the material intended to be extracted.

- In this concern like dissolve like, which means polar solvents dissolve polar materials and vice versa.

3-Temperature:

Increasing the temperature leads to increase in the solubility and diffusion of extracted materials so increase the extraction rate. Also increasing temperature decreases the viscosity so increase the extraction rate.

4-Agitation:

Agitation leads to increase the rate of extraction. It increases the diffusion to the bulk of the solvent so increase rate of extraction. This is because agitation cause dilution of the stagnant layer around the particles and also it prevents sedimentation of particles.

$$dM/dt = K' A (Cs - C)/ b$$

where: -

M = amount to be extracted

dM/ dt = Rate of extraction

T=time

A = Area of solid - liquid interface

b = effective thickness of liquid film surrounding the particle

K' = diffusion coefficient

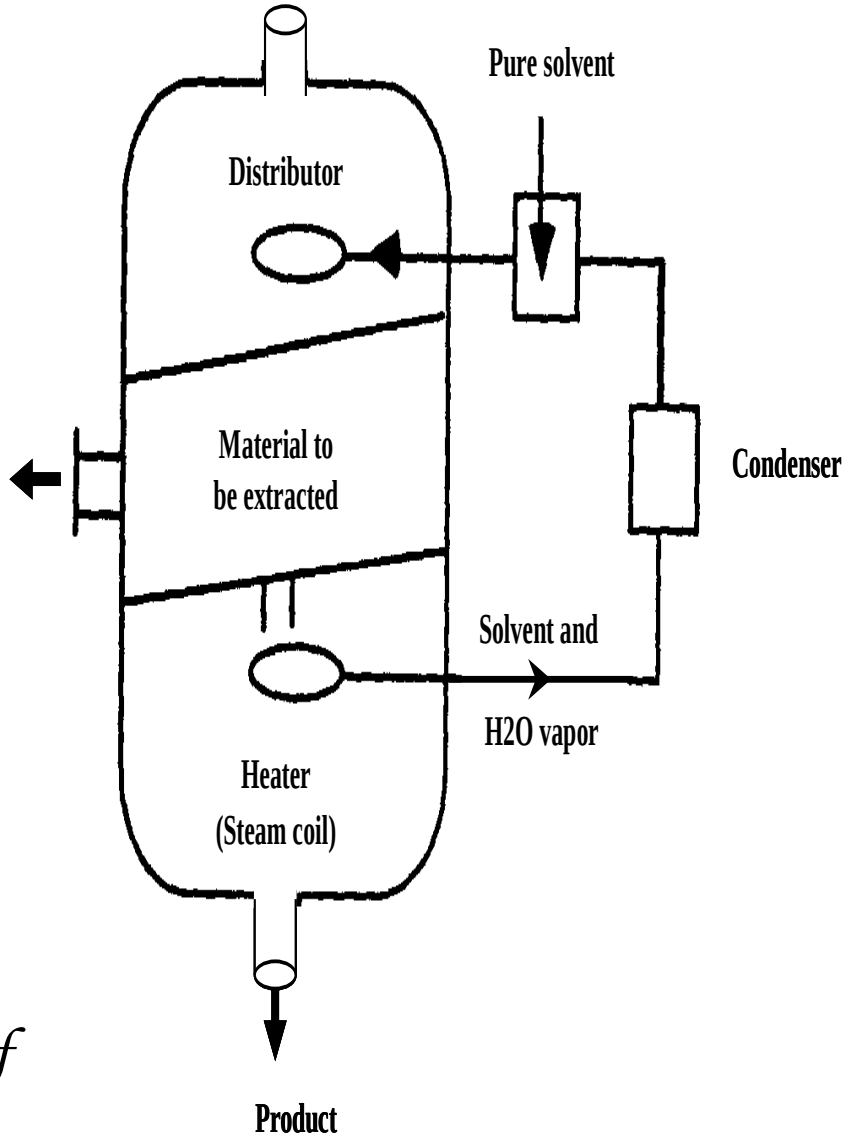
C = Concentration of solute in the bulk of solution at time t

Cs = Concentration of saturated solution in contact with the particle.

Extraction Equipment (leaching)

1-Batch plant Extractor:

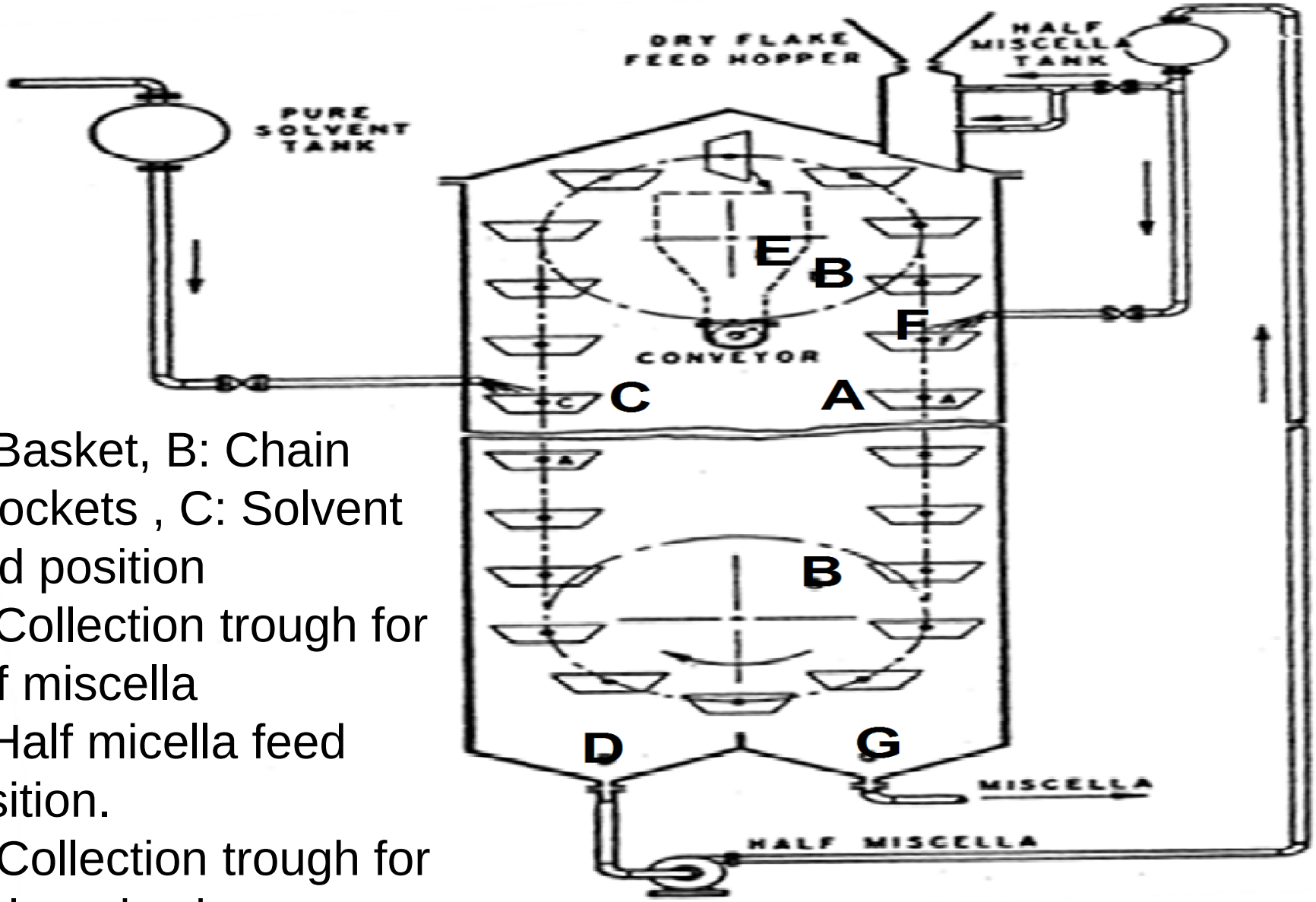
- It consists of a cylinder divided into two sections the upper in filled with the material to be extracted, and the solvent is sprayed from distributor. The solution is evaporated by steam coil and the produced vapor will pass through a condenser and recycled again via the distributer.



Uses: *Extraction of oils from seeds.*

Disadvantages: *It needs large amount of solvent and it is time consuming*

2-Bollman Extractor: (Basket extraction)



A: Basket, B: Chain sprockets , C: Solvent feed position
D: Collection trough for half miscella
F: Half micella feed position.
G: Collection trough for final production

It consists of : -

- - vertical chamber in which a number of baskets (A) with perforated metal bottom .
- The baskets (A) are carried on an endless chain running over two sprocket-wheels (B).
- As the buckets rise at the end of their travel, fresh solvent is added into each bucket at C, so that the solvent percolates down through the seeds in the rest of the buckets in the rising column (counter-current extraction)
- The resulting dilute solution collects in the bottom of the apparatus at D. It is called half miscella which will circulate by the pump and sprayed over the fresh feed at F.
- Another part of this half-micella is introduced into the feed hopper for preliminary extraction and damping of the feed.
- At the top of their travel, the buckets are inverted, and the exhausted material is discharged into a chute E, from which it is remove by a screw conveyer.

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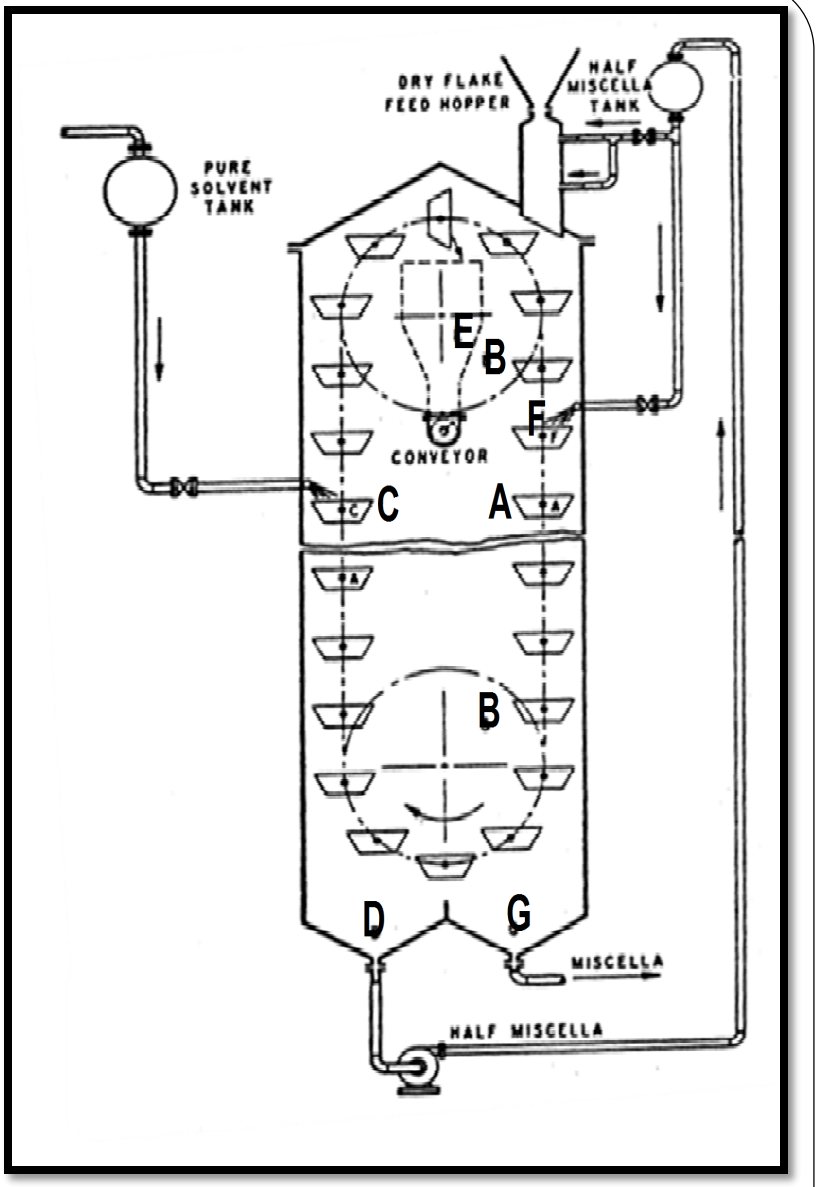
- Then, the buckets come under a feed hopper and are filled with fresh material.
- As they descend they are sprayed with the dilute solution(half micella) near the top of the column at F, Which then percolates down through the puckets in the descending column (co-current extraction).
- Then it collects in the bottom at G as the fully concentrated miscella which is withdrawn as the final product.
- The extraction operation is in part counter- current (in the ascending column of buckets) and in part co-current (in the descending column of buckets) .
- Counter – current extraction is more effective and is desirable.



Uses

Extraction of oils from cellular materials especially edible oils from seeds

3- Roto-cel Extractor:

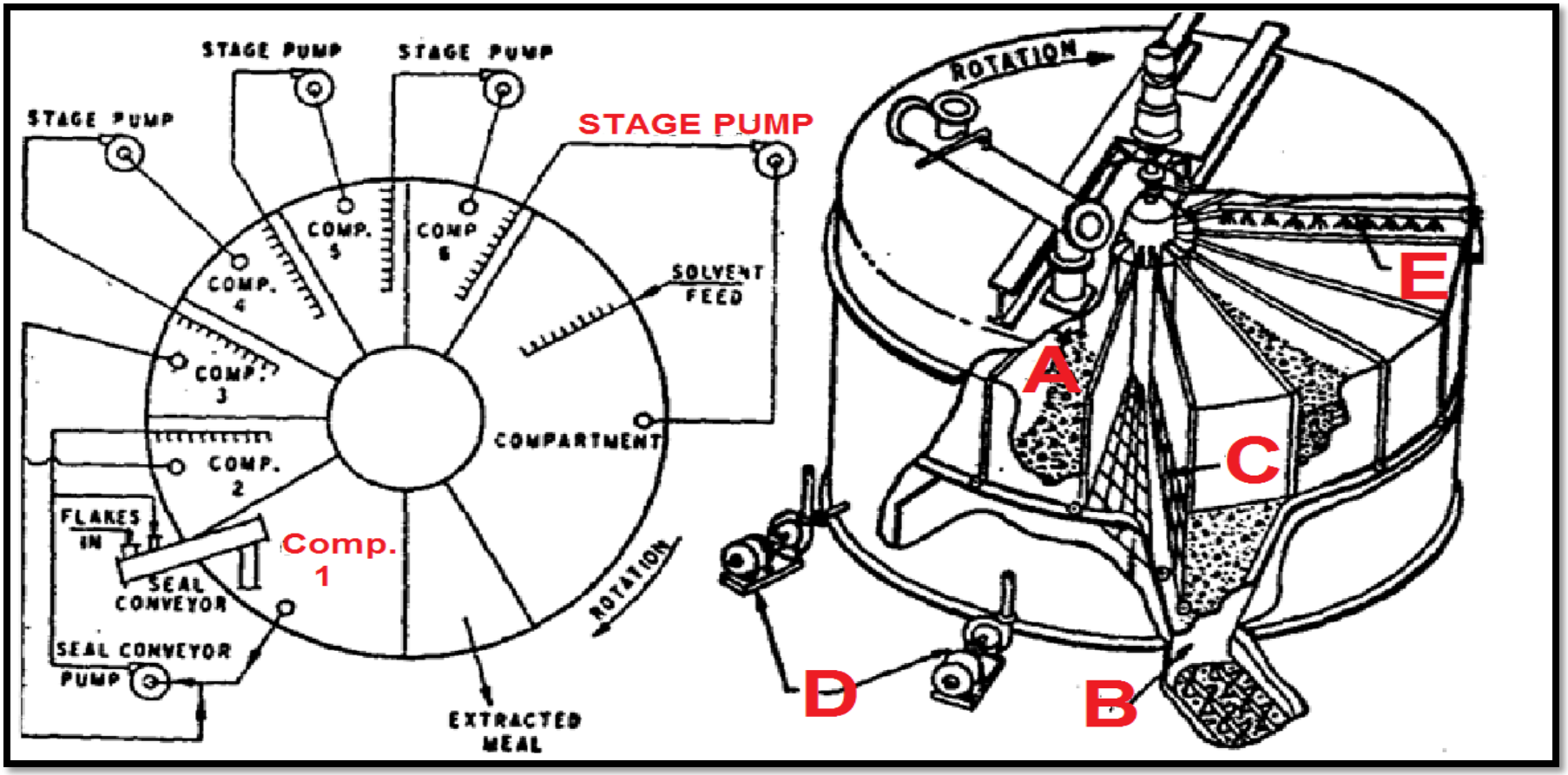


Diagram of operation of Rotocel extractor. (Blow Knox)

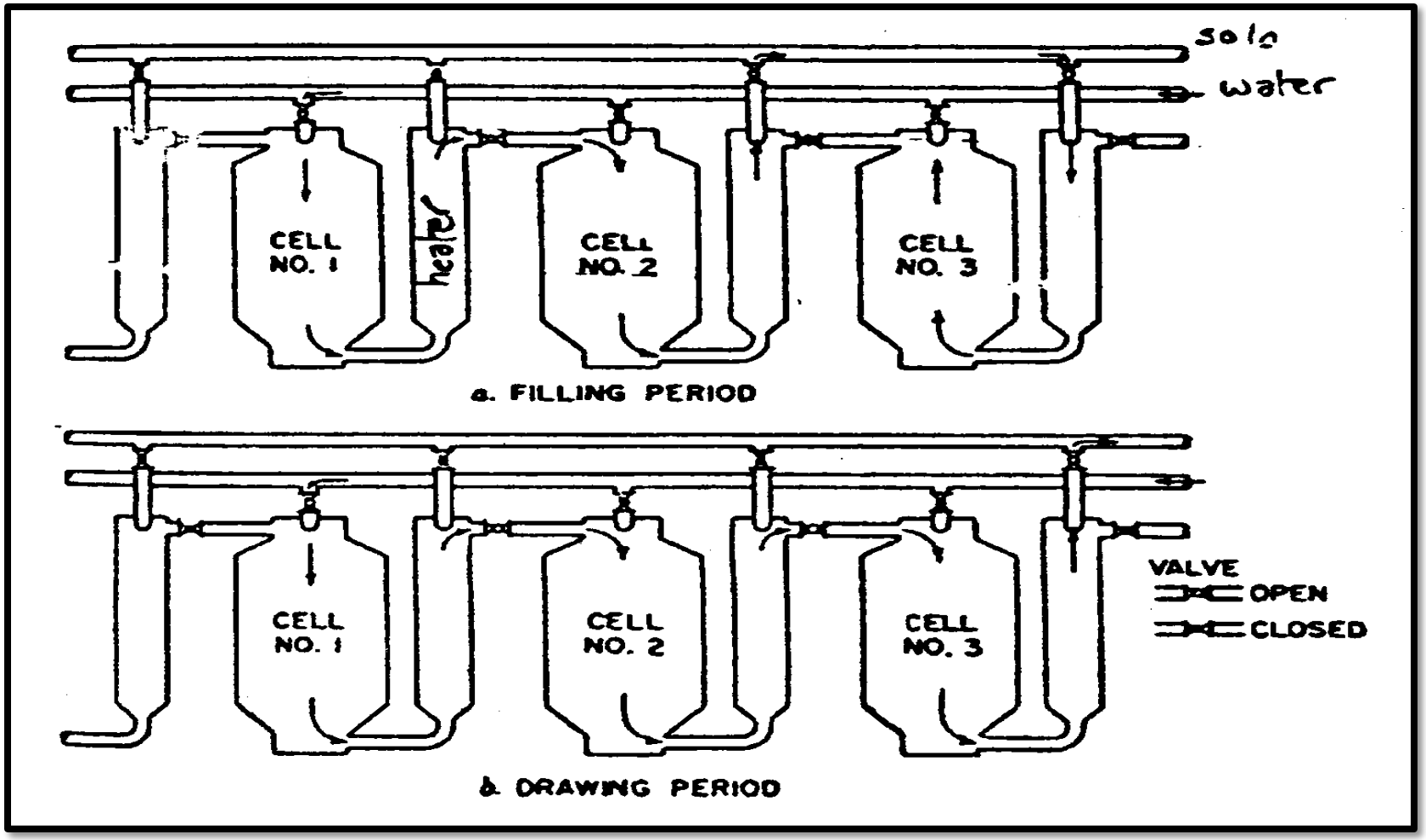
Rotocel - seed extractor : A , feed opening; B, discharge opening; C, hinged bottom of cells; D, stage pumps; E. sprays (Blaw-Knox)

- - It consists of a short cylinder. Its axis vertical, enclosed in a vapor tight housing.
- - This cylinder is divided into a considerable number of wedge shaped compartments hinged - perforated bottoms.
- - As the cylinder rotates on its vertical axis a given compartment first comes under a chute A where it is filled with material and then passes on to be treated with solvent in various stages of concentration.
- The cycle of the feed is opposite in direction to the cycle of the solvent. This is to make the fresh solvent extract the remains from the mark and to make the half micella in contact with the fresh powder to concentrate it as a final micella.
- After extraction is completed, the cell passes over a discharge chute B where the hinged bottom C drops and discharges the exhausted material.
- There is a series of pumps - called stage pumps D, which pumps the solvent out of the compartment at one position and discharge it into the compartment at the previous position through sprays E.
- This gives a true counter - current extraction.
- The solvent most widely used is hexane. Chlorinated solvents such as trichloroethylene .

Disadvantages:

- The cost of such solvents in too high to make them practical.
- The danger of explosion of hexane or a chlorinated solvent or the danger of excessive loss of solvent makes it necessary that the extractor be in a completely vapor tight housing. This complicates all problems of feed and discharge

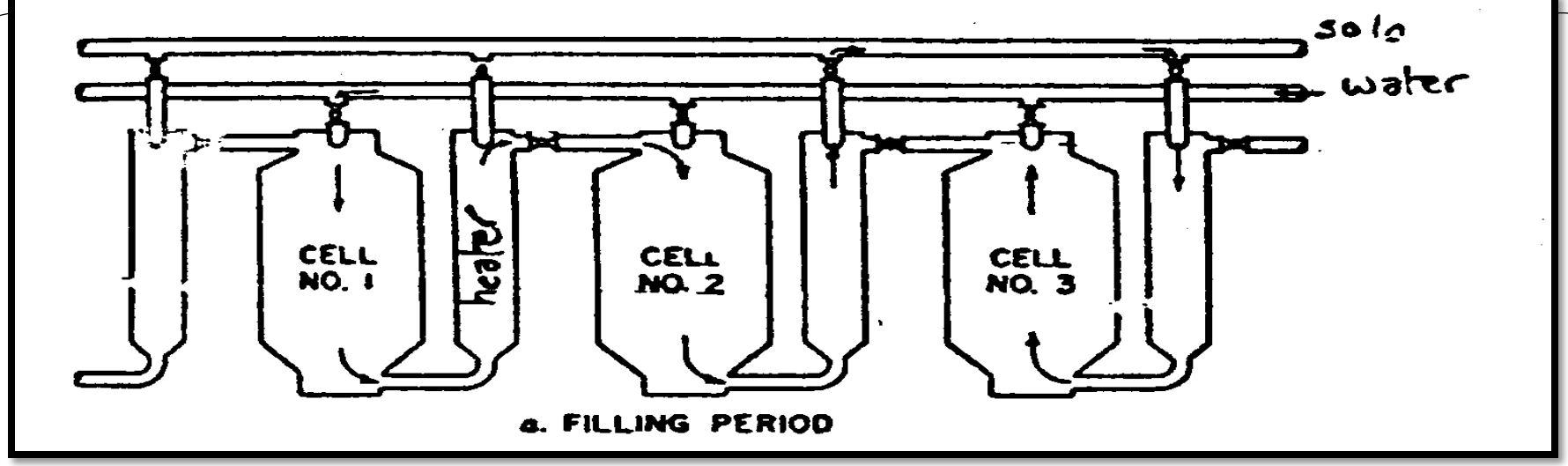
4- Fixed - bed or Robert diffusion battery



Uses:

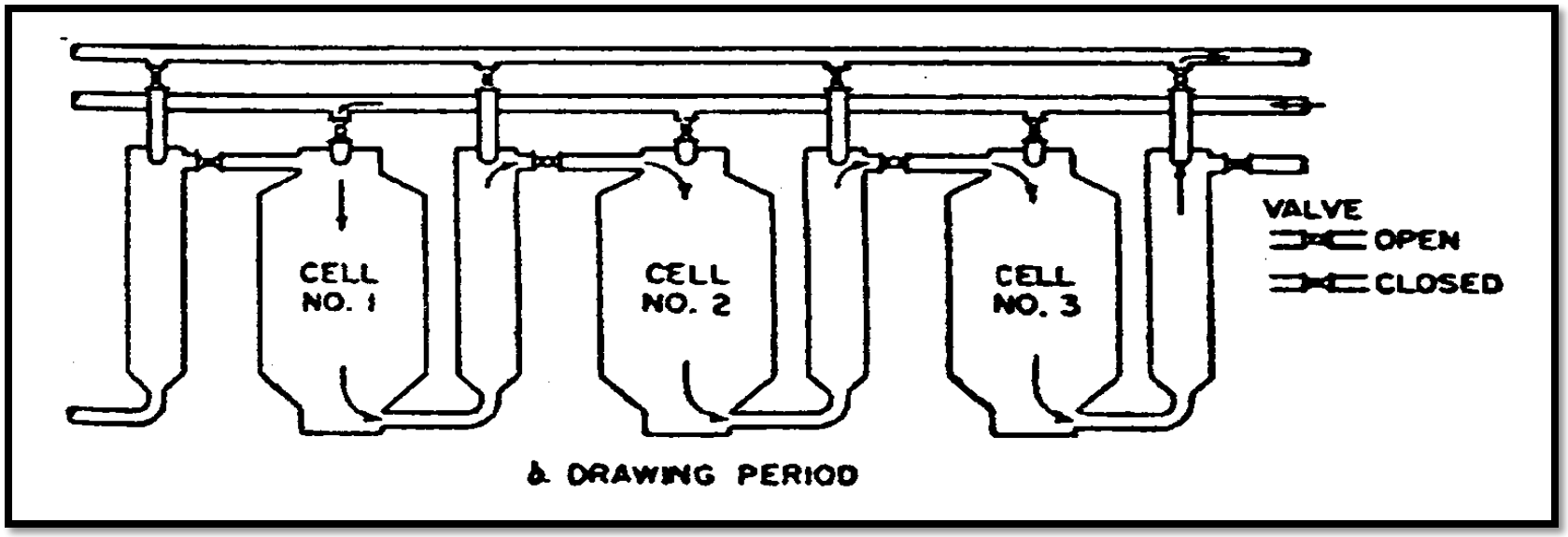
For leaching intermediate solids.
Used for the extraction of tanning extracts from tanbark.
For the extraction of certain pharmaceuticals from barks and from seeds .

- - It consists of a raw of vessels filled with the material to be extracted and through which water flows in series.
- The piping is so arranged that the fresh water comes in contact with the most nearly extracted material and the strongest solution leaves from contact with, the fresh material.
- For every vessel (or cell), there is a heater, because the diffusion process takes place more rapidly at higher temperature.
- Two main headers (tubes) are necessary. One handles water and the other handles solution and for every cell there must be 3 valves.
- In the figure, the valves that are open are shown as circles 0 and the valves that are closed are shown in solid black,



In the above Fig. a: (filling period).

- Cell 1 is nearly exhausted and cell 3 has just been charged.
- Water is introduced into cell 1 and flows down through the cell, up through the heater, down through cell 2, and up through its heater.
- It would not be convenient to pass the solution down through cell 3 because of the air which would be entrapped and the charge is cold, therefore, additional heating is desirable.
- Consequently, the liquid flows from the heater of cell 2 through the solution line, down through the heater of cell 3, and up through cell 3.



The valves are changed to the position shown in Fig b (Drawing period).

- Liquid now flows down through cell 3 up through its heater and out to the process.

The operation shown in Fig b is continued until cell 1 is completely extracted.

By this time another cell to the right of these shown has been filled, cell 1 is dumped, water is introduced to cell 2 and the process continued

Liquid -liquid extraction

It has many important applications:

(1) Purification of uranium fuel and recovery of spent fuel element in the nuclear power industry.

(2) Recovery of copper from acid leach liquors and subsequent electro-wining from these liquors.

(3) Separation of aromatics from kerosin to increase its burning quality and prevent smokes.

(4) Separation of aromatics from naphthalin and paraffin to improve temperature - viscosity characteristics of lubricating oils.

(5) Extraction of phenol from coal tar liquors.

(6) Purification of penicillin from impurities after the fermentation process.

(7) Production of anhydrous acetic acid by removing moisture.

(8) Obtaining relatively pure compounds such as benzene, toluene and xylene from catalytically produced reformates in the oil industry.

Requirements for liquid - liquid – extraction:

1- Selectivity of the solvent

2- Differential solubility of the two liquids in the solvent .

3- The solvent is immiscible with the liquid mixture

Liquid - liquid extraction takes place in three stages:

1- The immiscible phases come in contact with each other.

2- Separation of the mixture

3- Solvent recovery (distillation)

Extraction in many ways is complementary to distillation and is preferable in the following cases.

(1) Where distillation would require excessive amounts of heat, e.g., when the relative volatility is near unity

(2) When the formation of azotropes limits the degree of separation obtainable in distillation.

(3) When heating must be avoided.

(4) When the components to be separated are quite different in nature.

The extraction operation may be either a physical operation or a chemical operation.

Chemical operations have been classified by Hanson as follows:

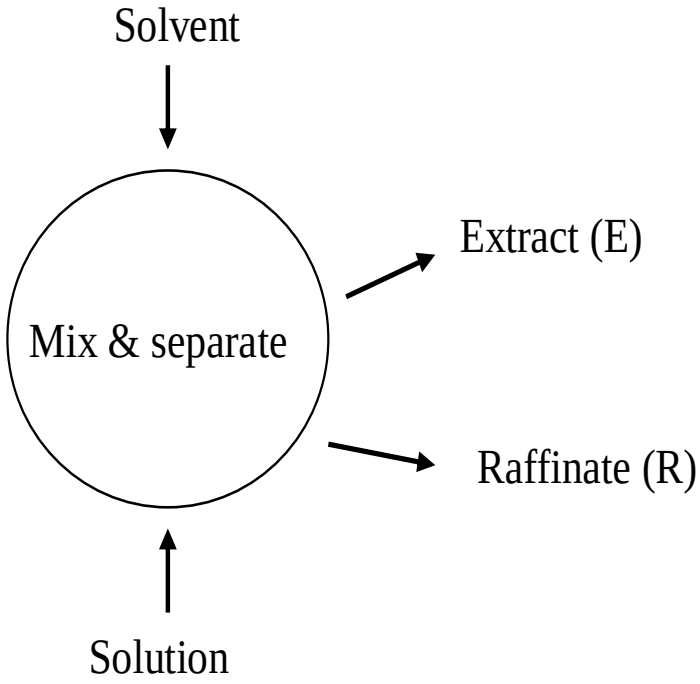
(1) Those involving cation exchange, e.g., extraction of metals by carboxylic acids

(2) Those involving anion exchange, e.g., extraction of anions involving a metal with amines.

(3) Those involving the formation of an additive compound ,e.g., extraction with neutral organophosphorus compounds, an important operation of this type is the purification of uranium from the nitrate with tri-n- butyl phosphate.

Extraction may be carried out either as a batch or as a continuous process

(1) Single stage batch process:

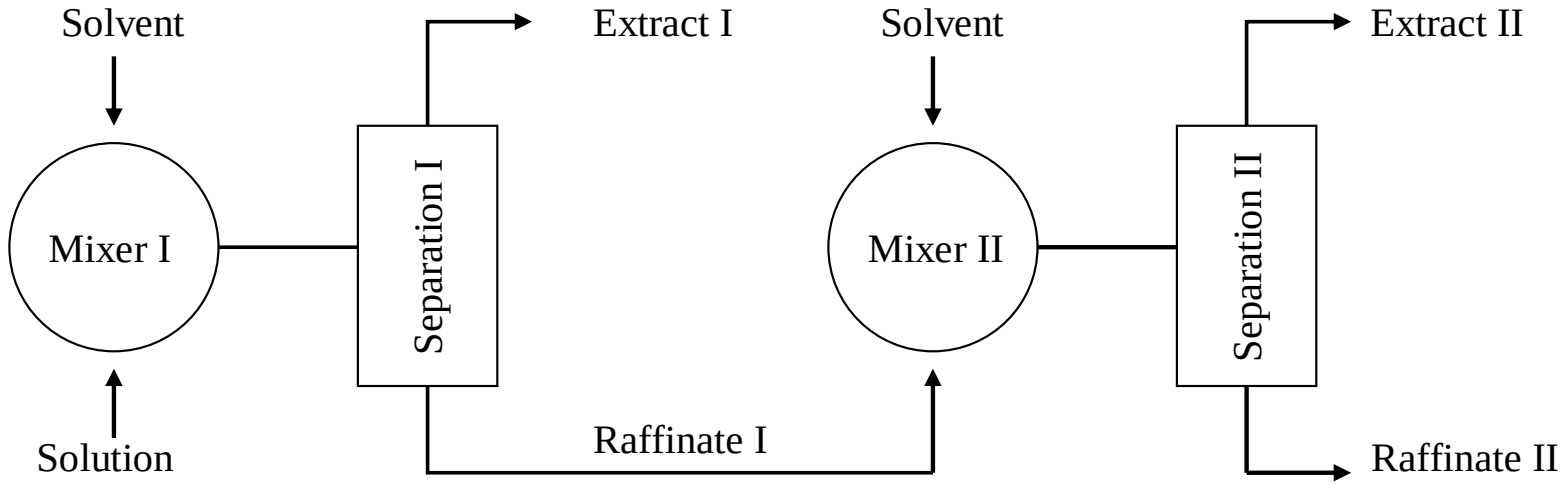


The solvent and solution are mixed together and then allowed to separate into the two phases:

a) **The Extract E** containing the required solute in the added solvent.

b) **The Raffinate R** the weaker solution with some associated solvent. With this simple arrangement mixing and separation occur in the same vessel.

(2) Continuous two-stage operation:



The mixers & separation are shown as separate vessels

Selection of solvent is very important :

When solvent is added to solution, one of the following processes occur,

(1) A homogenous solution is formed thus the solvent is not suitable

(2) The solvent may be completely immiscible with the initial solvent .The solvent in this case is ideal.

3) The solvent may be partially immiscible with the original solvent forming one pair of partially miscible liquid which is not good for extraction.

4) The new solvent may lead to formation of two or three pairs of partially miscible liquid.

EQUIPMENT

(A) Stage - wise equipment:

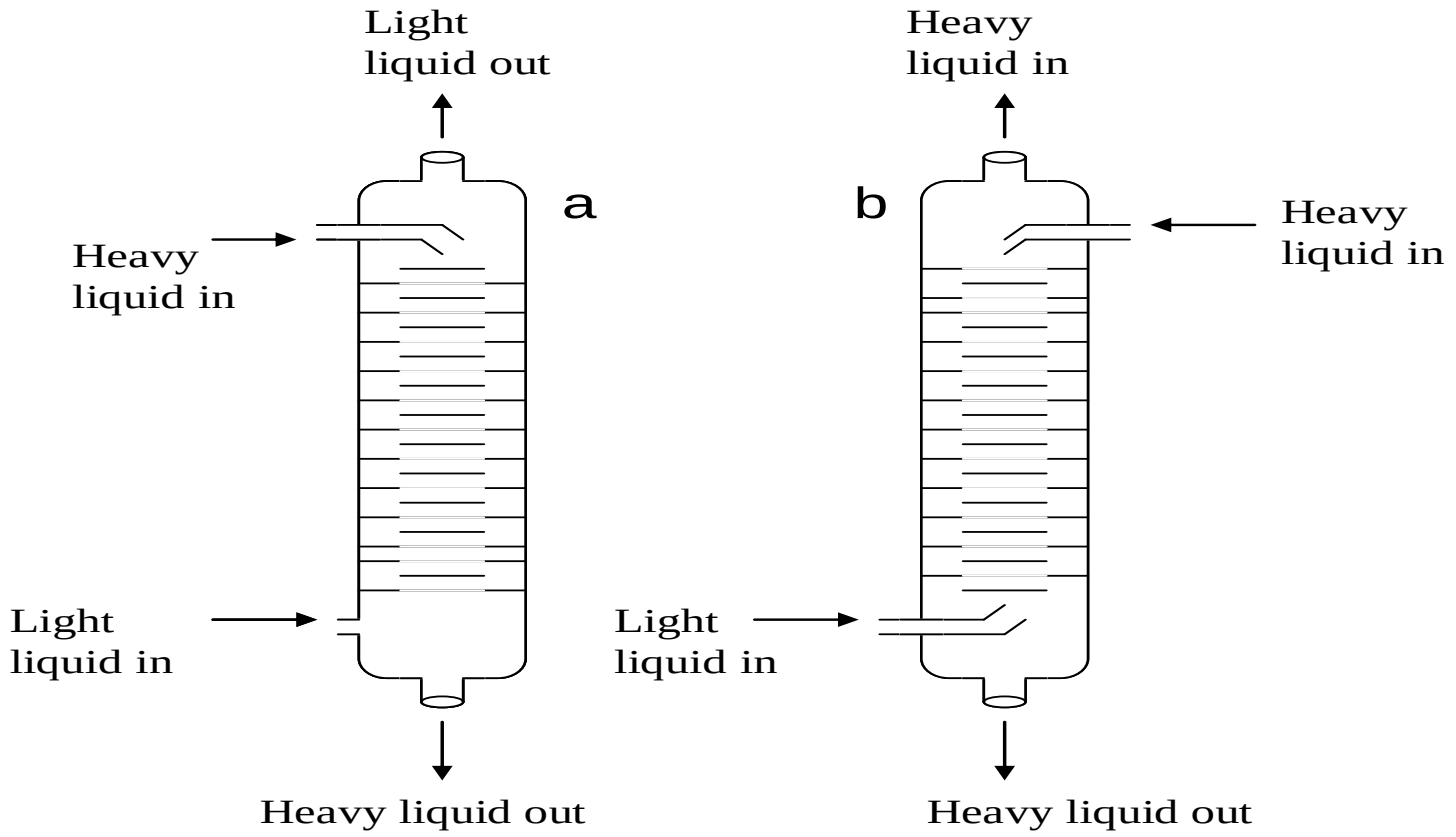
This equipment includes a series of physical stages in which the phases are mixed and separated.

(1) The mixer- settler

- In this unit the solution and solvent are mixed by some form of agitator in the mixer, then transferred to the settler where the two phases are separated to give an extract and a raffinate.
- The separation is gravity controlled and the liquid densities and the form of the dispersion are important parameters.
- Disadvantage: formation of a layer of an emulsion between the two immiscible liquids.

(2) Baffle – plate column

- Simple cylindrical columns provided with baffles to direct the flow of the dispersed phase.
- The efficiency of each plate is very low.
- The baffles can be positioned very close together (75- 150 mm) thus it is possible to obtain several theoretical stages in a reasonable height.



(3) The scheible column

- One of the difficulties in using packed columns is that redispersion of the liquids after each stage is very poor.

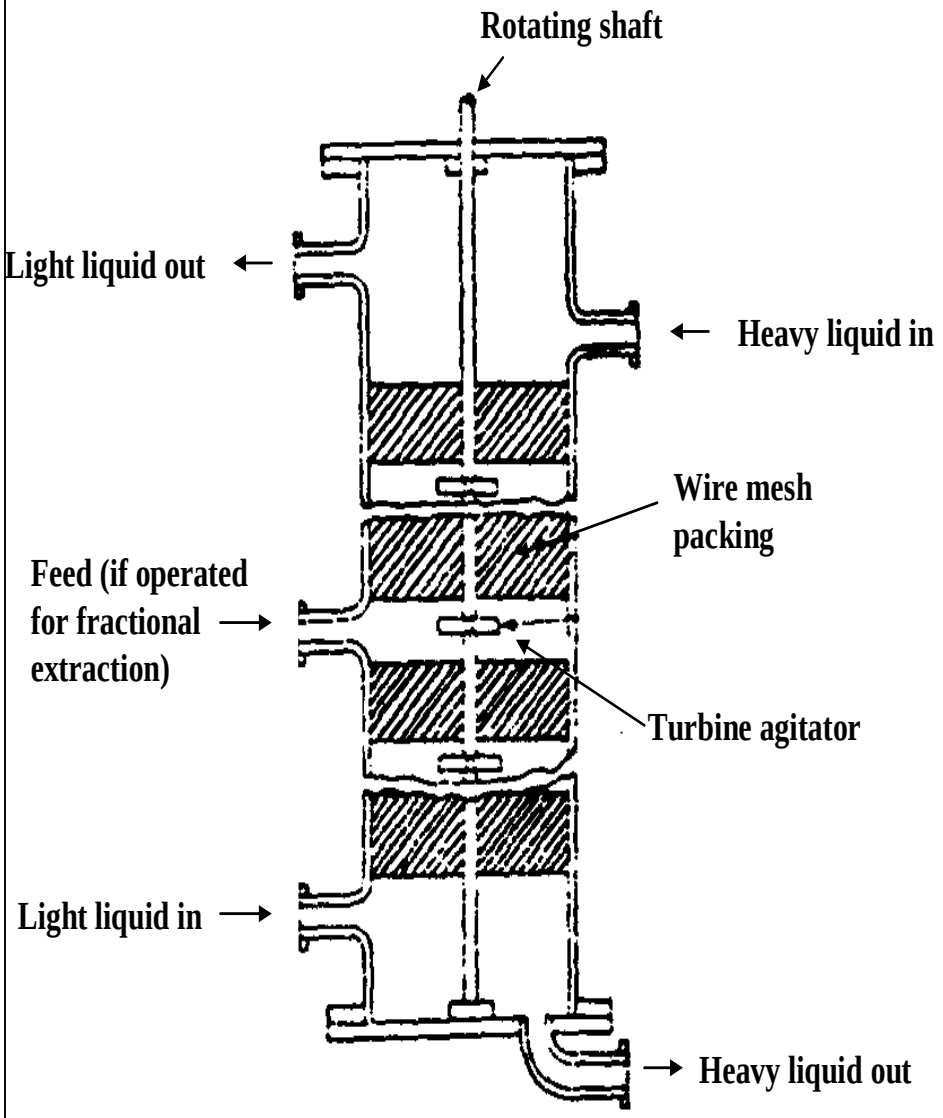
- To overcome this problem, Scheibel introduced a unit in which a series of agitators is mounted on a central rotating shaft.

- Between the agitators is fitted a wire mesh section which successfully breaks up any formed emulsions.

-**Advantages of Scheibel column:**

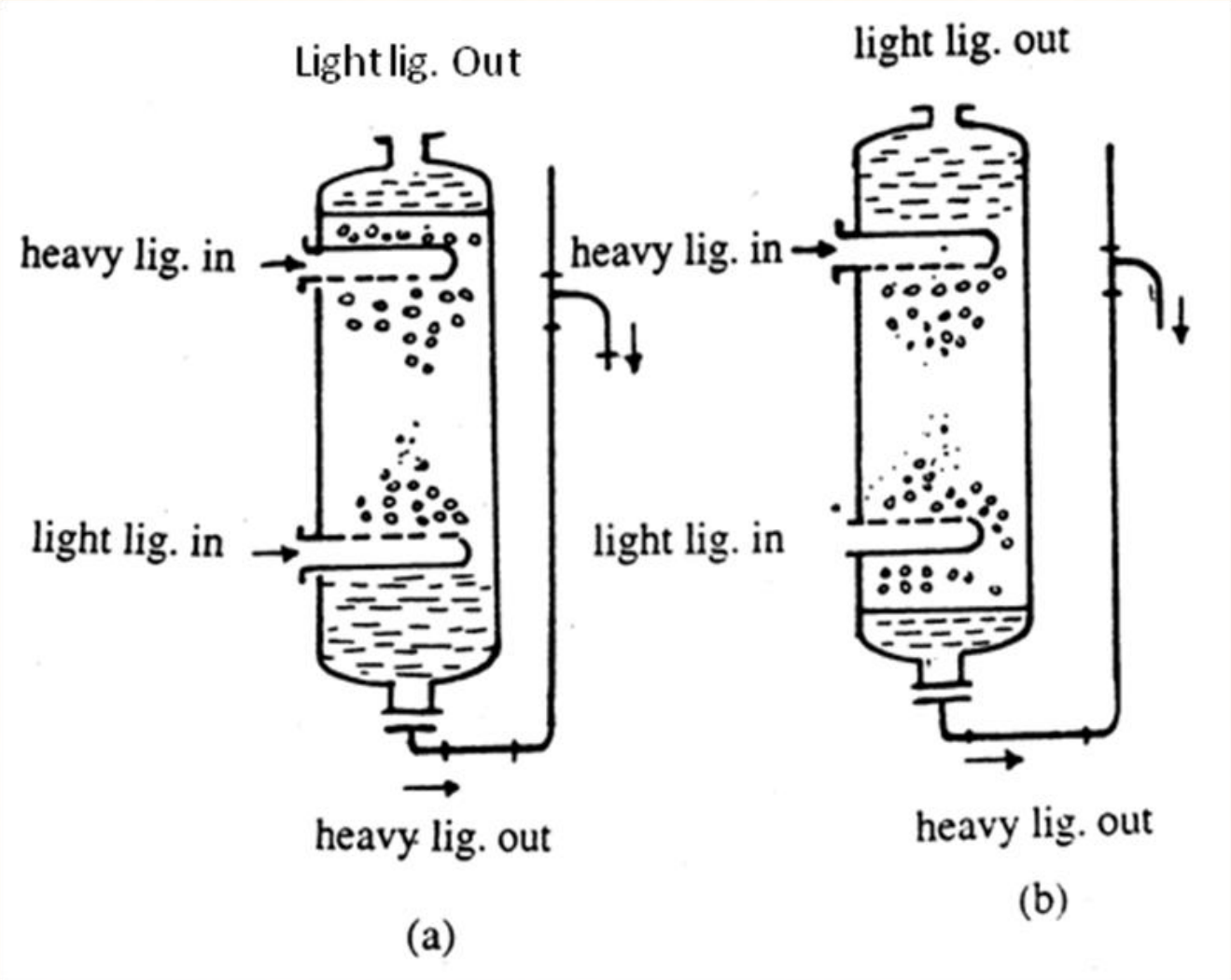
(1)- Redispersion of liquids is better because of the presence of agitators.

(2)- Breaking up any emulsion formed by the wire mesh.



(B) Differential contact equipment:

(1) spray column or spray towers:



- Two methods of operating spray columns. Either the light or heavy liquid phase may be dispersed in (a) the light phase enters from a distributor at the bottom of the column and the droplets rise through the heavier phase, finally coalescing to form a liquid - liquid interface at the top of the tower.

In (b) the heavier phase is dispersed in this case and the inter phase is held at the bottom of the tower.

- Disadvantages:

Although they are simple in construction, they are inefficient because :

(1) Considerable recirculation of the continuous phase takes place thus true counter current flow is not maintained and up to 6 mm may be required to obtain one theoretical stage.

(2) There is very little turbulence in the continuous phase and lack of interface renewal and appreciable axial mixing resulting in poor performance.

Because the droplets of the dispersed phase rise or fall through the continuous phase under the influence of gravity , there is a limit to the amount of dispersed phase that can pass through the tower for any given flow rate of continuous phase.

For example in figure (a) any additional light phase fed to the bottom in excess of that which can pass upwards under the influence of gravity, will be rejected from the bottom and the tower is said to be **flooded**.

(2) Packed columns

The packing increases the interfacial area and increases the mass transfer rate compared with those obtained with spray columns.

(3) Centrifugal extractors

Podbielniak extractor:

- The driving force for mixing and separation is the centrifugal force.

- The heavier liquid is introduced at (E) passes through channel (F) and enter the rotating plate assembly at (G).

- The whole rotating element is turning at 2000 - 5000 rpm which gives centrifugal force that drive the heavier liquid out through the perforations in the plates to collect in space (H), taken by channels (J) , leave by connections (K).

- The lighter liquid is introduced at (L) passes through channels (M), discharged in space (H).

- Since the heavier liquid is being driven outwards by centrifugal force, it displaces the lighter liquid which flows downward through the perforated plates, collects in space (N) and passes out to channels (0) to leave at connection (p).

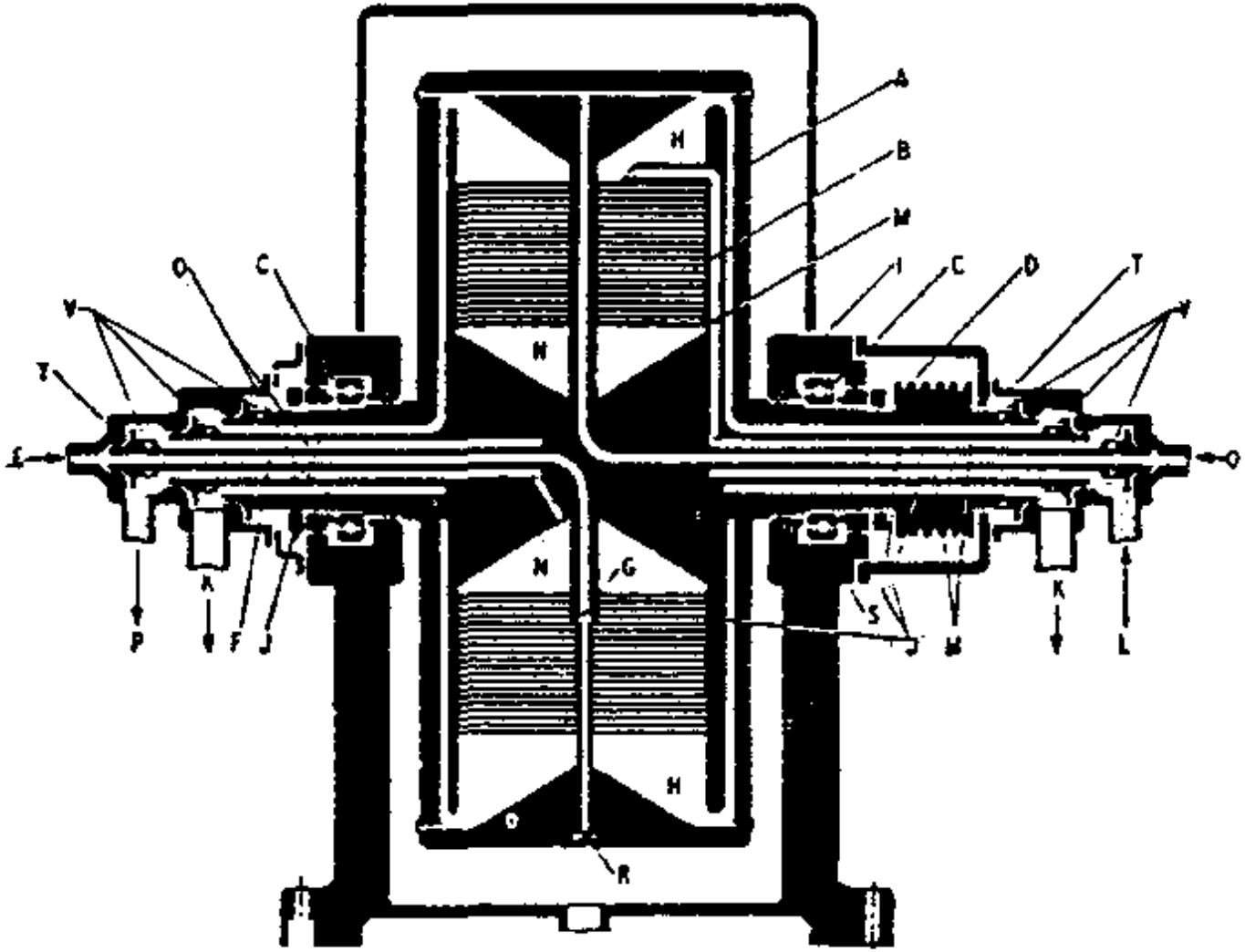
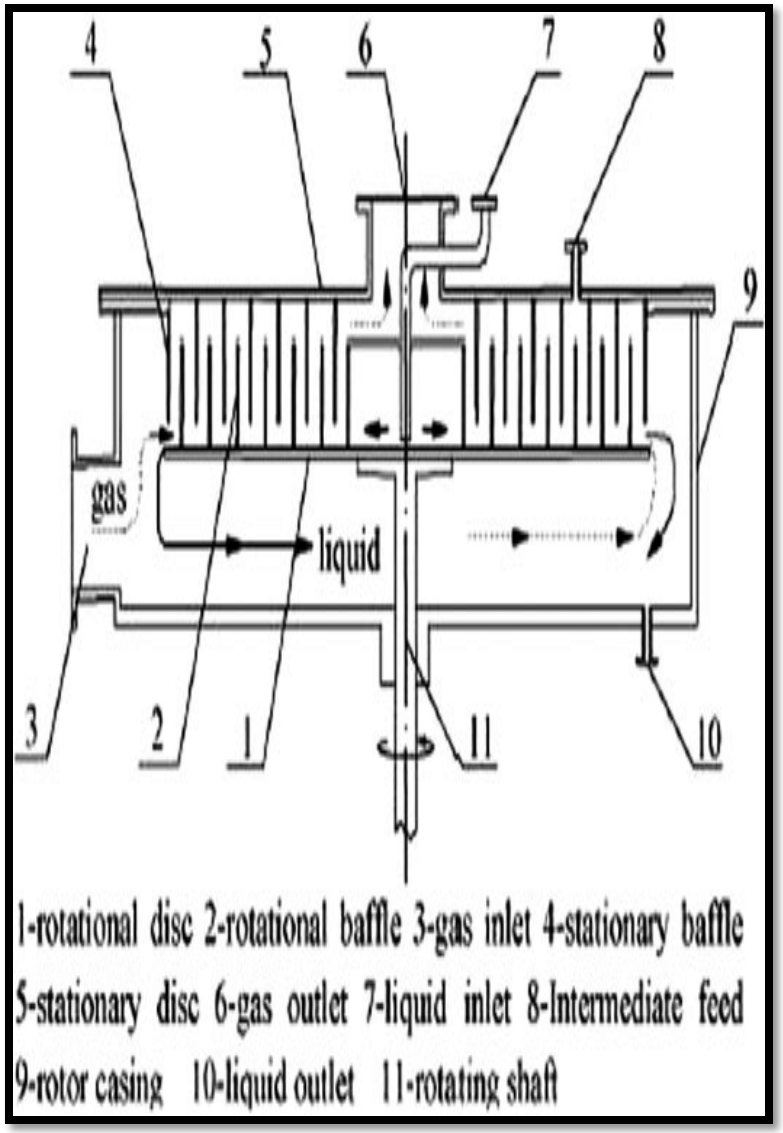
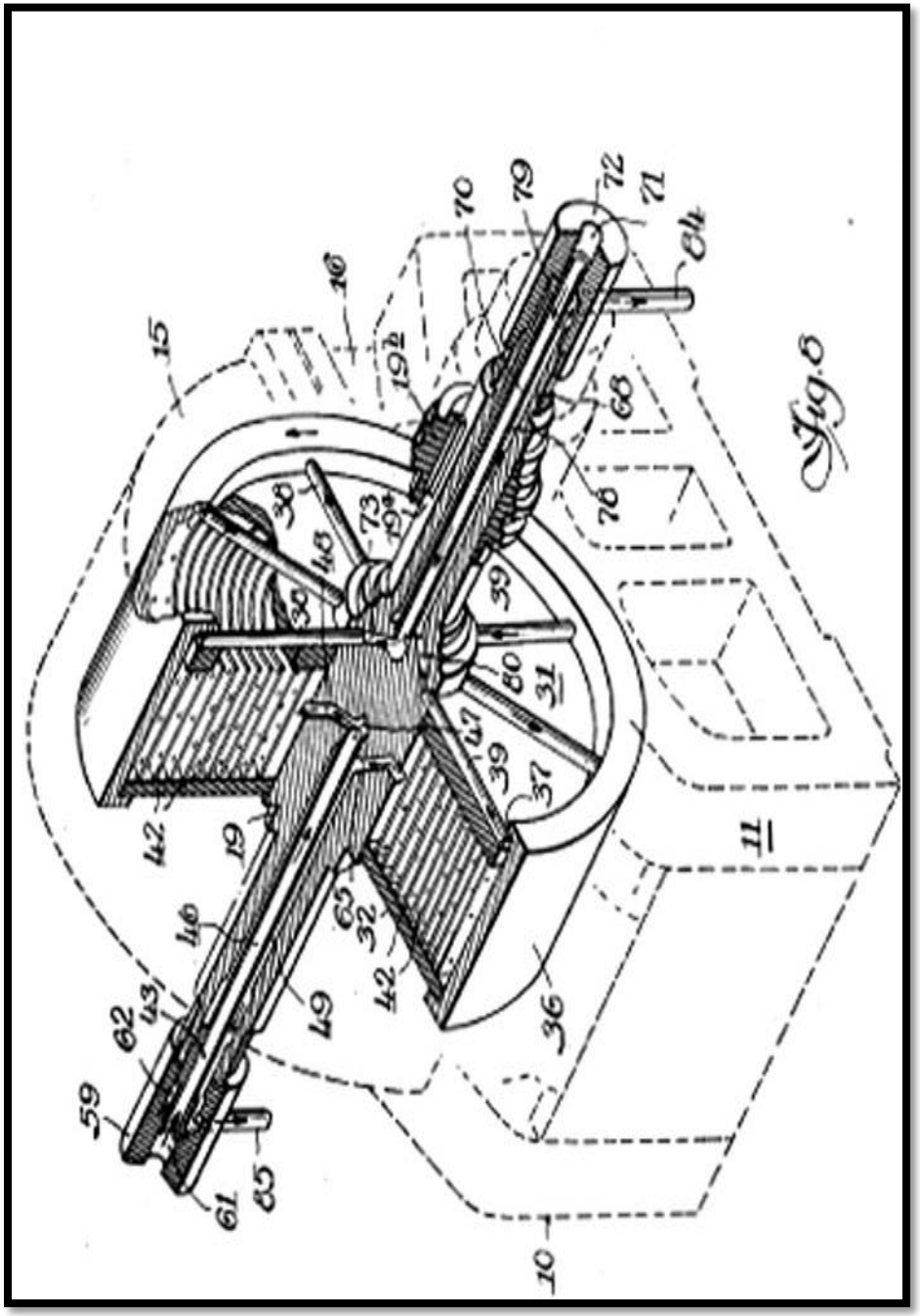


Fig. Podbielniak extractor: A rotor; B. perforated plates ; C. main bearing; D, drive pulley; E. inlet for heavy liquid; f. G. ports for heavy liquid, H, collecting space for heavy liquid J. exit ports for heavy liquid, K. heavy – liquid exit L. light liquid inlet M , N. O. path of light liquid P light liquid exit Q, inlet for wash water R , c1eanout plugs.S, main stationary housing T, stationary member carrying pipe connections V , mechanical seals.





podbielniak extractor

- Advantages:

1- The contact between the two liquids is intimate, so that extraction of a soluble constituent from one liquid into the other is quite complete.

2- The residence time of the liquid is short (few seconds) which could lead to possible extraction of unstable substances.

** It is used where high performance phase separation and counter current extraction or washing in the one unit are required.

- Important applications of podbielniak extractor are:

1- Removal of acid sludges from hydrocarbons

2- Hydrogen peroxide extraction

3- Sulphonate soap and antibiotic extraction

4- Extraction of rare earths such as uranium and vanadium from leach liquors.

5- Washing of refined edible oils