

11

LEACHING

11.1 INTRODUCTION

Leaching is one of the oldest operations in chemical industries which involves the use of a solvent to remove a solute from a solid mixture. Though originally it was referred to the percolation of liquid through a bed of solids, it is now used to refer the operations by other contacting means also. *Lixiviation* is used for the leaching of alkali from wood ashes. *Decoction* refers to the operation where the solvent at its boiling is used. Whenever the solute material is present largely on the surface of an insoluble solid and is merely washed off by the solvent, the operation is called *elutriation or elution*.

It is one of the most important operations in metallurgical industries for the extraction of metals from ores of Al, Ni, Co, Mn and Zn. It is also used for the extraction of sugar from sugar beets with hot water, extraction of oil from oil seeds using organic solvents, removal of tannin from various tree barks by leaching with water, preparation of tea and coffee and extraction of many pharmaceutical products from plant roots and leaves.

The success of this operation depends on the proper preparation of the given solid. Depending on the nature of solid, the solid is crushed and ground to desired size to accelerate the leaching action. For example, a certain copper takes about 6 hours if crushed to – 60 mesh size and about 5 days for a size of 6 mm. Gold is sparsely distributed in its ore. Hence it is crushed to – 100 mesh size to have an effective leaching. Sugar beets are cut into thin wedge shaped slices called cassettes before leaching to enable the solvent water to reach the individual plant cells. In the manufacture of pharmaceutical products from plants they are dried in order to rupture the cell walls so that solvent can reach the solute easily. Vegetable seeds when used for the extraction of oil are crushed to a size of 0.15 to 0.5 mm to enable easier extraction. However, when the solid is present on the surface, no grinding or crushing is necessary and the particles can be washed directly. To summarize, the leaching action depends on:

- The nature of solid/cell structure.

- Diffusion of solute from the material to surface and then to the bulk of the solution.
- Particle size and its distribution.
- Solubility of solute in solvent and the temperature of operation.

11.2 UNSTEADY STATE OPERATION

These operations are carried out batchwise or semibatchwise.

11.2.1 In Place (in-situ) Leaching

This operation is also called *solution mining* which refers to the percolation leaching of minerals in place at a mine, by circulation of the solvent over and through the ore bed. This technique is adopted for the leaching of low-grade copper. In these operations, the solvent/reagent is injected continuously through one set of pipes drilled down to the ore and the resulting solution is pumped out through another set of pipes. Alternatively, the solvent/reagent can be pumped into the ore bed intermittently and withdrawn through the same well. In this technique crushing and grinding of ore are avoided. In place leaching also called in-situ leaching is shown in Fig. 11.1.

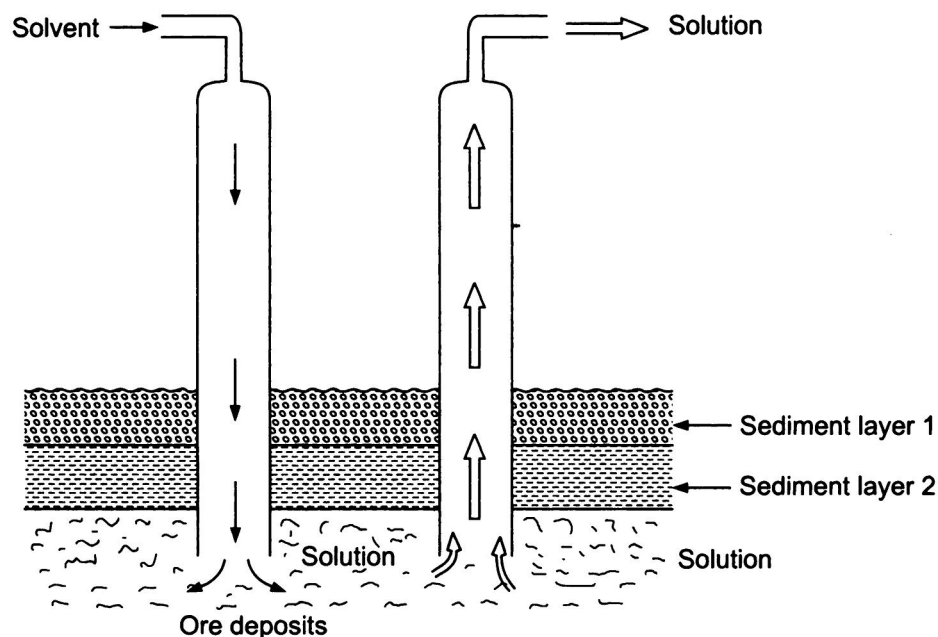


Fig. 11.1 In situ leaching.

11.2.2 Heap Leaching

Low-grade ores can be easily leached by this technique where the ore is gathered as a heap upon impervious ground. The leach liquor is pumped over the ore, which percolates through the heap and collected as it drains from the heap. This technique is used for the extraction of copper and uranium from their low grade ores. Heap leaching is shown in Fig. 11.2.

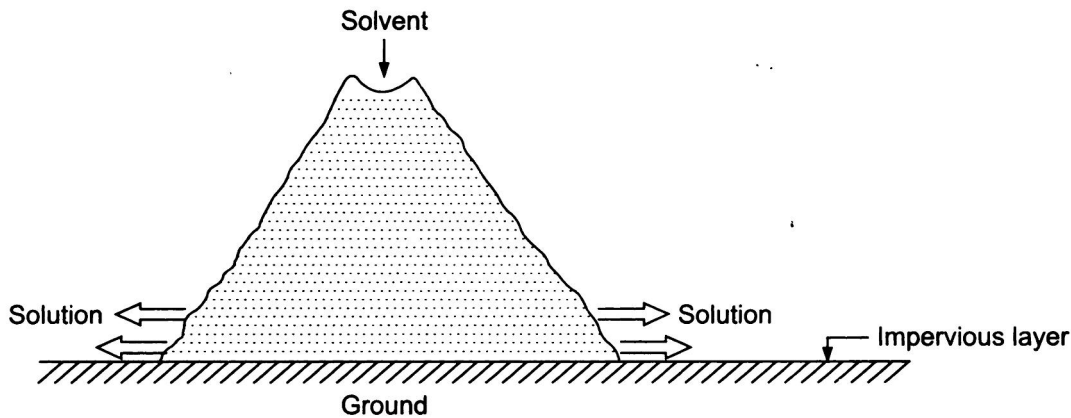


Fig. 11.2 Heap leaching.

11.2.3 Percolation Tank

Whenever small tanks are to be used, they can be made of metal or wood. The solid particles to be leached, rest on a false bottom which could be made of wood strips and may be covered by a coconut matting and a tightly stretched canvas filter cloth. The leach liquor flows to a collection pipe leading from the bottom of the tank. A very large percolation tanks are made of reinforced concrete and lined with lead or bituminous mastic. Small tanks may be provided with side doors near the bottom for removing the leached solid while the large tanks are emptied by excavating from the top. It is always preferable to fill the tanks with particles of uniform size so that voids will be more and the pressure drop required for flow of leaching liquid is least. This also leads to uniform leaching of individual particles.

For these operations the crushed solids may be filled in the tank initially and then the solvent is allowed to enter in. The solid and solvent may remain in contact with each other for a specified amount of time and then drained. During the process, if necessary, the liquid can also be circulated through the bed. The liquid can also be allowed to enter in continuously and also drained continuously. The liquid from the exit can also be recirculated, if necessary. The flow of liquid could either be downwards or upwards with proper distribution of liquid. Percolation tank is shown in Fig. 11.3.

11.2.4 Countercurrent Contact

At times, one is interested in getting a strong solution, which can be obtained by a countercurrent operation. This arrangement, also called *Shanks system*, contains number of tanks as shown in Fig. 11.4. The number of tanks generally vary from 6 to 16. In a typical system with 8 tanks at a particular time, tank 8 is empty and tanks 1 to 7 contain solids. Fresh solvent enters tank 1, where the solid has spent maximum amount of time and the material in tank 2, 3, 4, 5, 6 and 7 have progressively spent lesser time. The material in 7th tank has spent the least amount of time. The solution withdrawn from the 7th tank has the maximum solute concentration because the solution comes after contact with fresh solids. The solution withdrawn from tank 1 goes to tank 2, from tank 2 to tank 3, ..., tank 6

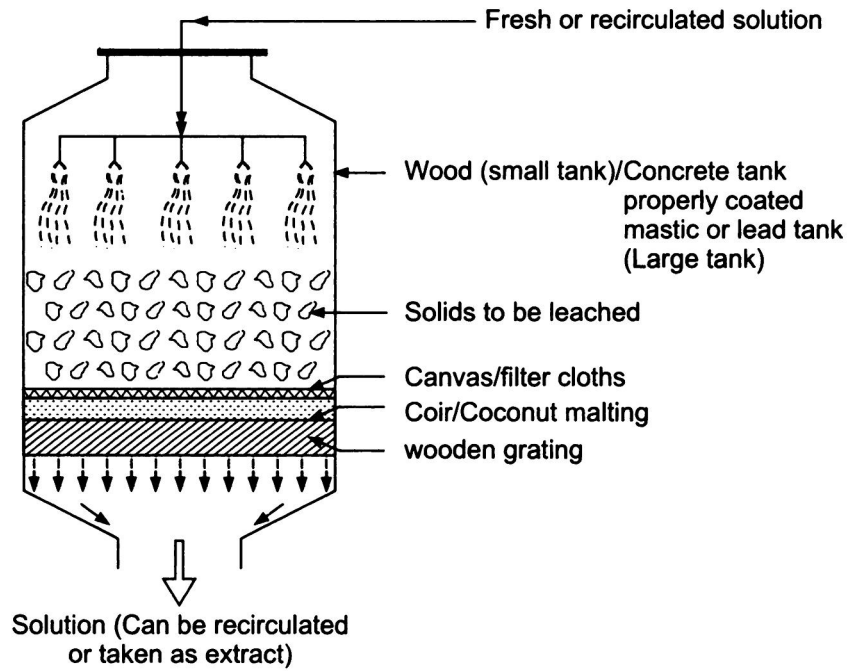


Fig. 11.3 Percolation tank.

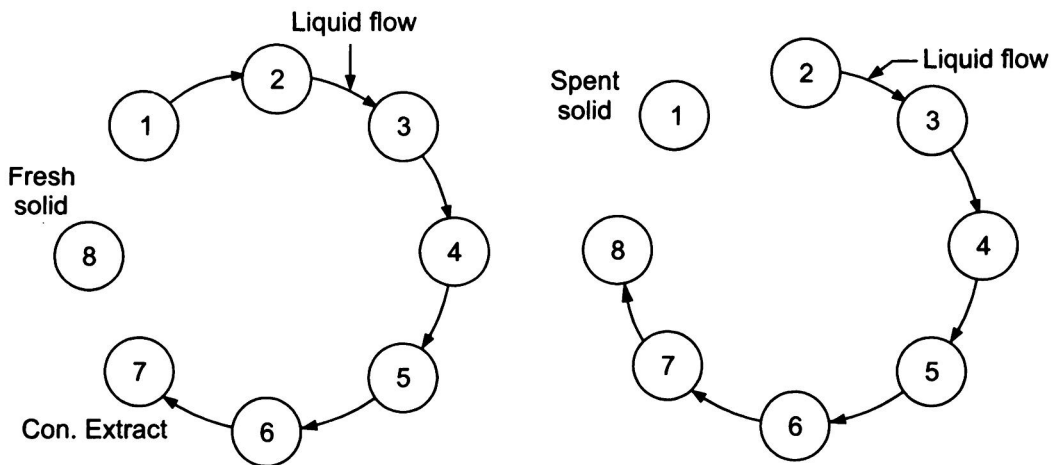


Fig. 11.4 Countercurrent system—shanks system.

to tank 7. The leached solid is discarded from tank 1 and fresh solid is now added in tank 8. The solution is transferred from tank 7 to 8, 6 to 7, 5 to 6, ..., 2 to 3. Here the fresh solvent is added in tank 2, and the solid from tank 2 is finally discarded. The solution now obtained from tank '8' will have maximum solute concentration. Tank 1 which is now empty will be loaded with a fresh batch of solids. This is nothing but advancing the tanks by one. The operation is continued in this manner by keeping successive tanks as the first tank in which the fresh solvent enters. The solids move counter currently to liquid flow.

11.2.5 Percolations in Closed Vessels

At times the pressure drop for flow of liquids by gravity is high or the solvent is highly volatile. Under such circumstances the liquid is pumped through the bed of

solids in vessels called diffusers. The main advantage of these units is the prevention of evaporation losses of solvent, when they are operated above the boiling point of solvent (e.g. leaching of tannins using water at 120°C, 345 kN/m²).

11.2.6 Filter-Press Leaching

When the solids are in finely divided form, percolation tanks are not suitable. Under such circumstances, solids can be filtered and leached in the filter press by pumping the solvent through the press cake. This is also a common feature while washing the filtered cakes.

11.2.7 Agitated Vessels

These are either vertical or horizontal closed cylindrical vessels with power-driven paddles or stirrers on vertical or horizontal shafts. They have a provision at the bottom for the withdrawal of leach solution at the end of the operation. In some of the designs, the horizontal drum is the extraction vessel, and the solid and liquid are tumbled about inside by rotation of the drum on rollers. They are operated on batch basis and each one is a single leaching stage. They can also be used in series for a multistage operation. For the leaching of finely divided solids, Pachuca tank is used. This finds extensive use in metallurgical industries. These tanks are constructed with wood, metal or concrete and lined with suitable material depending on the nature of leaching liquid. Agitation is accomplished by air lift. The bubbles rising through the central tube cause the upward flow of liquid and suspended solid in the tube and hence circulation of the mixture. Conventional mechanical agitators are also used for this purpose. Once the desired leaching is achieved, the agitation is stopped, the solids are allowed to settle and the clear supernatant liquid is decanted by siphoning over the top of the tank or by withdrawal through discharge pipes placed at appropriate level in the side of the tank. Whenever, the solids settled form a compressible sludge, the solution retained will be more and generally the last traces of solute in such cases are recovered in countercurrent manner.

11.2.8 Features of Percolation and Agitation Techniques

If a solid is in the form of big lumps, the question that arises is whether one should go in for percolation technique or agitation-settling technique. The problem is quite complicated due to the diverse leaching characteristics of the various solids and the value of solute. However, the following points are worth considering.

Though fine grinding is more costly and provides more rapid and possibly more thorough leaching, the quantity of liquid associated with the settled solid is very large. Hence, one may have to use large quantity of solvent to recover as much solute as possible. The composited extract thus obtained could be dilute. Coarsely ground particles, on the other hand, leach more slowly and possibly less thoroughly and may retain lesser quantity of solution. They may also require lesser

washing and hence the extract could be a concentrated one due to the use of lesser quantity of solvent.

Practical results have shown that leaching in an agitated vessel is more effective than by percolation for a fibrous solid like sugar cane.

Hence, one may have to decide based on the economy and the ease of operation.

11.3 STEADY STATE OPERATIONS

They are classified as stagewise or continuous contact operations. Stagewise equipment are sometimes assembled in multiple units to produce multistage effects, whereas, continuous contact equipment provide the equivalent of many stages in a single unit. Some of the solids may also require grinding in order to make the soluble portions accessible to the leaching solvents. In fact, wet grinding is an operation during which some leaching could be accomplished. For example, 50 to 75% of the soluble gold may be dissolved by grinding the ore in the presence of cyanide solution. Castor oil is also extracted suitably in an attrition mill with solvent.

11.3.1 Agitated Vessels

Finely ground solids which can be readily suspended in liquids by agitation can be handled in agitated vessels. These must be arranged for continuous flow of both liquid and solid in and out of the tank. Care must be taken to ensure that no accumulation of solid occurs. Due to thorough mixing, equilibrium is always there between the solid and liquid. The agitated vessels discussed earlier can also be used.

The average holding time can be estimated both for solids and liquids separately in an agitated vessel by dividing the vessel contents by the rate of flow of solids and liquids. The average holding time should be adequate to provide the required leaching action. Short circuiting is a disadvantage encountered which can be eliminated by passing the solid-liquid mixture through a series of smaller agitated vessels such that the cumulative holding time is the required leach time. The effluent from continuous agitators are sent to a filter for separating liquid from solid upon which the solid may be washed free of dissolved solids, or to a series of thickeners for countercurrent washing.

11.3.2 Thickeners

There are mechanical devices which are meant for increasing the ratio of solid to liquid in a suspension of finely-sized particles by settling and decanting, thus producing a thickened sludge and a clear supernatant liquid. They are generally installed before any filter to minimize the filtering costs. Since both effluents can be pumped and transported, thickeners are frequently used to wash leached solids and chemical precipitates free of adhering solution in a continuous multistage countercurrent arrangement and hence worth their use in leaching operations also.

The liquid content in the sludge varies from 15 to 75% and is greatly dependent on the nature of the solids and liquids and upon the time allowed for settling. This is shown in Fig. 11.5.

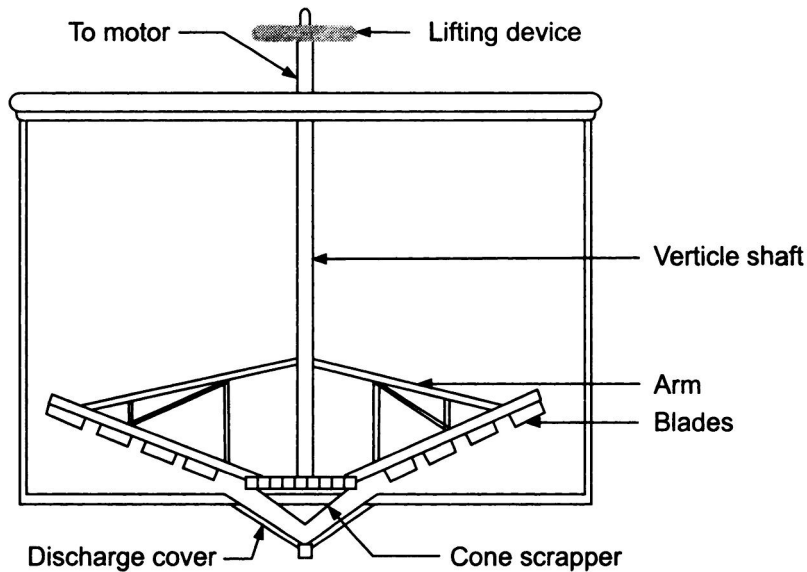


Fig. 11.5 Thickeners.

11.3.3 Continuous Countercurrent Decantation

It is an arrangement involving both the thickeners and agitators/grinders. The solids enter the first set of agitators/grinders and are mixed with overflow liquid from the 2nd thickener. Then the contents after through agitation/grinding enter the 1st thickener. The agitators along with thickener constitute the first stage. The sludge from the first thickener passes on to the 2nd thickener where it is mixed with overflow from the 3rd thickener and the sludge is then transferred to 3rd thickener where it is mixed with overflow liquid from 4th. Fresh solvent enters the last thickener. The overflow liquid taken out from the first thickener will have the maximum concentration of solute. If necessary the sludge from each stage can be thoroughly agitated with the solvent in order to effect better separation. This is shown in Fig. 11.6.

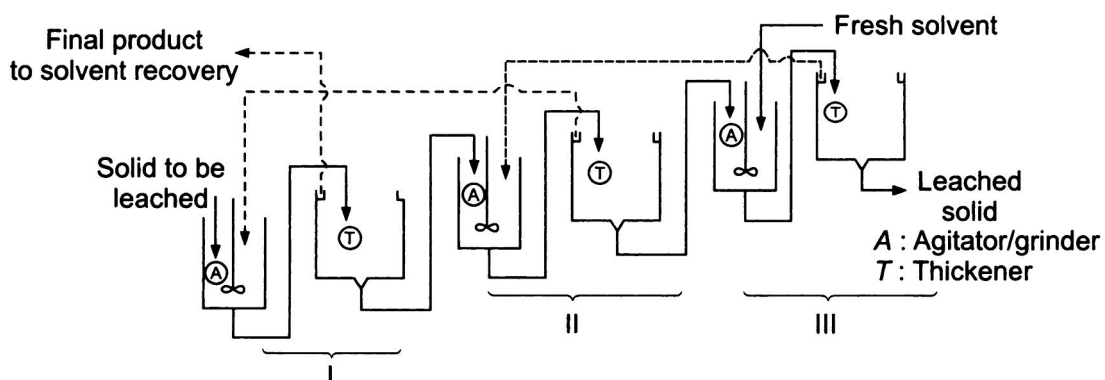


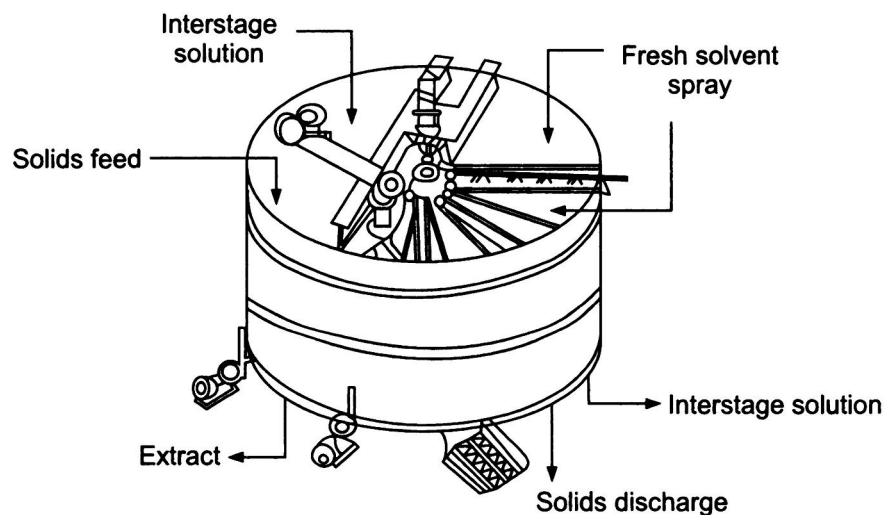
Fig. 11.6 Continuous countercurrent decantation.

11.3.4 Leaching of Vegetable Seeds

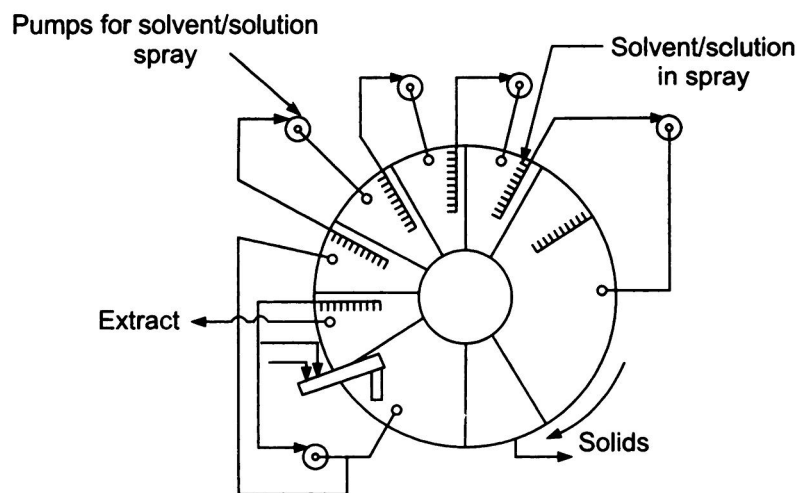
Soya beans, cotton seeds, rice bran and castor seeds are some of the products regularly leached with an organic solvent for removing the oil present in them. The process involves dehulling, precooking, adjustment of water content and flaking. In some cases solvent extraction of oil is preceded by mechanical expression of oil from oil seeds. Leaching solvents are generally petroleum fractions. Chlorinated hydrocarbons leave the residue meal a toxic one. The oil-solvent solution containing a small amount of finely divided suspended solids is called *miscella* and the leached solid is called *marc*.

11.3.4.1 Rotocel extractor

It is a modification of shanks system wherein the leaching tanks are continuously moved, permitting a continuous introduction and discharge of solids. It is shown in Fig. 11.7(a) and (b).



(a) Rotocel extractor (front view).



(b) Rotocel extractor (top view).

Fig. 11.7

It consists of a circular shell partitioned into several cells each fitted with a hinged screen bottom for supporting the solids. This shell slowly revolves above a stationary compartmented tank. As the rotor revolves, each cell passes in turn under the prepared solids feeder and then under a series of sprays by which the contents in each cell is periodically drenched with solvent for leaching. By the time one rotation is completed, when the leaching is expected to be completed, the leached solids of each cell are automatically dumped into one of the lower stationary compartments, from which they are continuously conveyed away. The solvent sprayed over each cell filled with solids, percolates downward through the solid and supporting screen into the appropriate compartment of the lower tank from which it is pumped to the next spray. The leaching is countercurrent, and the strongest solution comes from the cell which is filled with fresh solid. It is essential to maintain the equipment properly to ensure smooth operation. It is also enclosed in a vapour tight housing to prevent the escaping of solvent vapours.

11.3.4.2 Kennedy extractor

A schematic arrangement is shown in Fig. 11.8. It is a stagewise device, originally used for leaching tannins from tan bark. The solids are leached in a series of tubs and are pushed from one to next in the cascade by perforated paddles, while the solvent flows in countercurrent direction. Perforations in paddles permit drainage of liquid from solids between stages, and the solids are scrapped from each paddle as shown in Fig. 11.8. The number of tubs depends on the nature of solid, solvent and the level of extraction desired. Since it has a horizontal orientation, more floor space is required.

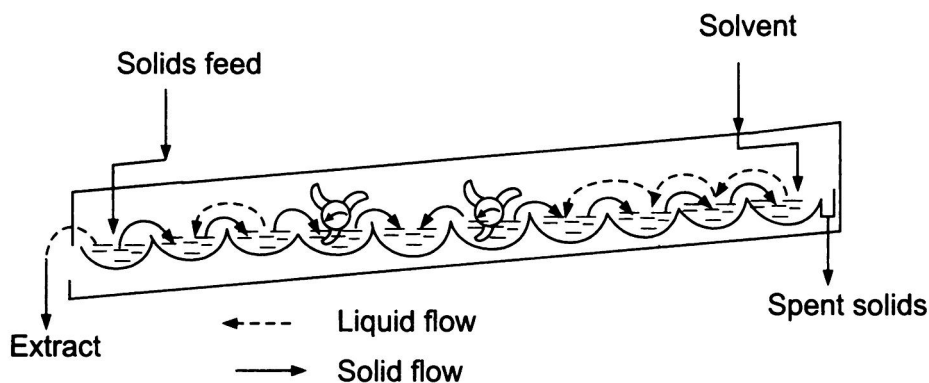


Fig. 11.8 Kennedy extractor.

11.3.4.3 Bollman extractor

It has a vertical orientation and has several perforated baskets attached to a chain conveyor for conveying solids. As the chain descends, the solids are leached in parallel flow by a dilute solvent – oil solution, called *half miscella*, pumped from the bottom of the vessel and sprayed over the baskets at the top. The liquid percolates through the solids from basket to basket and collects at the bottom as a final strong solution called *full miscella* and is withdrawn. On the ascent, the solids are leached countercurrently by a spray of fresh solvent and the product is called *half miscella*. A short drainage time is provided before leached solid in the baskets are dumped at the top. A schematic arrangement is shown in Fig. 11.9.

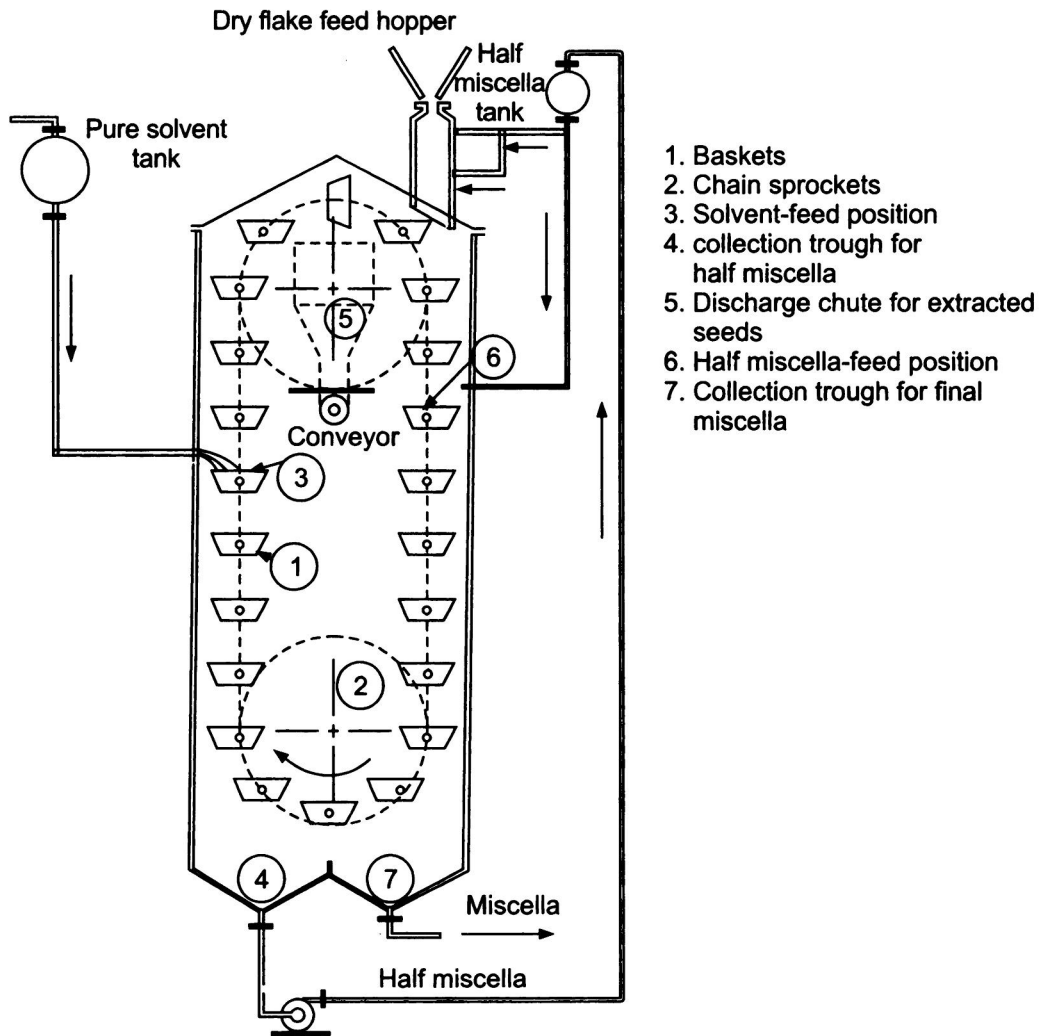


Fig. 11.9 Bollman extractor.

11.3.4.4 Continuous horizontal filter

A schematic arrangement of continuous horizontal filter is shown in Fig. 11.10. The filter in the form of a circular wheel is divided into a number of sectors and revolves in the horizontal plane. Here prepared seeds are slurried with solvent which has already been used for leaching, and the slurry is sent to the filter. The

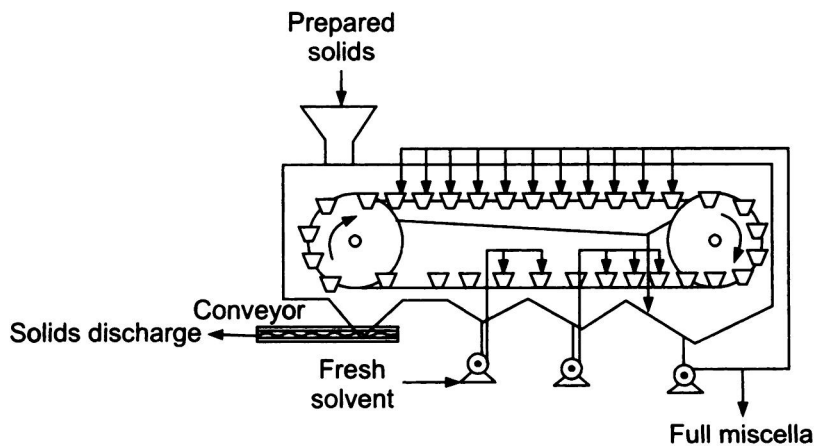


Fig. 11.10 Continuous horizontal filter.

first filtrate is passed again through the filter cake to remove finely divided solids (polishing) before being discharged as miscella. The principle behind the operation is quite similar to Rotocel extractor.

11.3.4.5 Recovery of oil

The recovery of solvent from both the miscella and leached solids is an essential feature in these operations. Recovery of oil in miscella is accomplished by evaporation of solvent and if necessary by further stripping in a tray column to remove the solvent-free oil. The oil in solid is removed by steaming and subsequent cooling. Vent gas from condensers can be sent to an absorber and scrubbed with petroleum white oil and the resulting mixture can be stripped to recover the solvent.

11.4 DEFINITIONS

Let, B = insoluble solid or inert solid (kg),

C = soluble solute (kg),

A = pure solvent (kg),

$x = \frac{C}{A + C}$; Weight fraction of solute in effluent solution (on B free basis)

$y = \frac{C}{A + C}$; Weight fraction of solute in the solid or slurry or sludge (on B free basis)

$N = \frac{B}{A + C}$; (in each phase)

The variation of N , x and y under different conditions are as follows:

(a) For a dry solid (free from solvent) $N = \frac{B}{C}$ ($\because A = 0$)

$$y = 1.0$$

(b) Solid free from solvent and solute $N = \infty$ ($\because A = 0; C = 0$)

(c) Pure solvent $x = 0, N = 0$ ($\because B = 0; C = 0$)

11.5 DIFFERENT TYPES OF EQUILIBRIUM DIAGRAMS

11.5.1 Type 1

A typical trend of N vs x , y and equilibrium relationship is shown in Fig. 11.11(a) characteristics of such systems are:

- Preferential adsorption of the solute occurs on solid.
- Solute is soluble in the solid B and distributes unequally between liquid and solid phases at equilibrium.
- Insufficient contact time between solute and solvent.
- EF is a tie line.

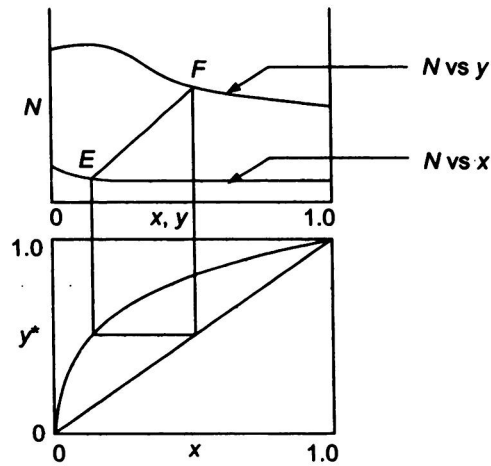


Fig. 11.11(a) Type I equilibrium.

11.5.2 Type II

A typical trend of N vs x , y and equilibrium relationship is shown in Fig. 11.11(b). The characteristics of such systems are:

- No adsorption of solute occurs.
- Solution withdrawn and the solution associated with the solid have the same composition.
- Tie lines are vertical.
- The distribution coefficient is unity.
- Solids are drained to the same extent at all solute concentrations and such a condition is known as *constant underflow condition*.
- No B is present in solution either dissolved or suspended.

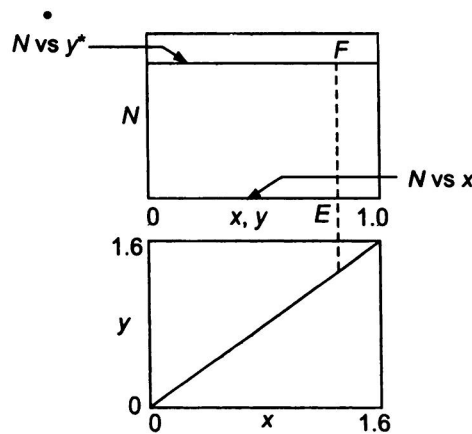


Fig. 11.11(b) Type II equilibrium.

11.5.3 Type III

A typical trend of N vs x , y and equilibrium relationship is shown in Fig. 11.11(c). The characteristics of such systems are:

- Solute C has a limited solubility x_S in solvent A and one can never have a clear (leach) solution stronger than x_S .
- Tie lines joining slurry and saturated solution converge as shown.
- Till the concentration of x_S is reached, the solution retained in the solid and

the clear solution have some concentration and hence the distribution coefficient is unity, i.e. up to the tie line FE. No adsorption of solute occurs.

- The tie lines to the right of FE indicate the same solute concentration in clear solution but a different solute concentration in slurry as indicated by points G, H.

In practice we come across situations which will fall in any one of the above three types.

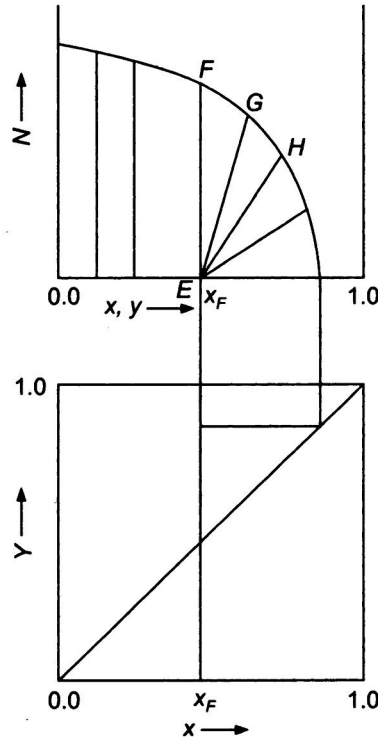


Fig. 11.11(c) Type III equilibrium.

11.6 SINGLE STAGE OPERATION

A typical single stage operation is shown in Fig. 11.2. The characteristics of various streams flowing into and out of the system are also shown. The flow rate of streams are on B free basis.

(All the streams are on absolute mass basis)

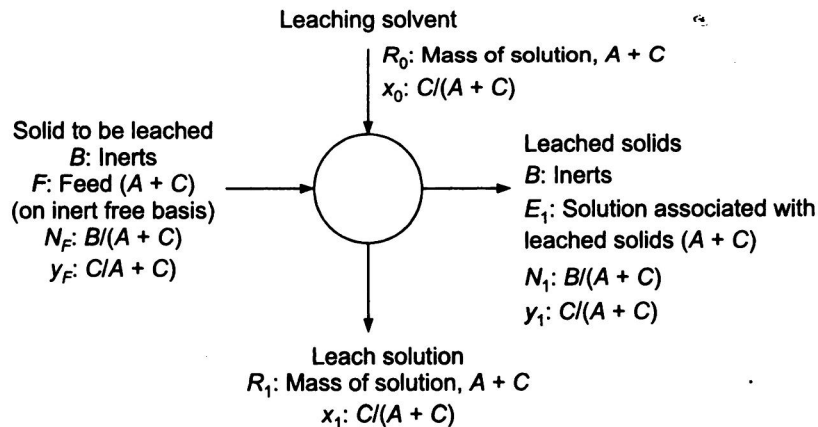


Fig. 11.12 Streams in a leaching operation.

$$N_F = \frac{B}{A + C} = \frac{B}{F}$$

$$N_1 = \frac{B}{A + C} = \frac{B}{E_1}$$

$$\therefore B = N_F \cdot F = E_1 N_1$$

Total material balance gives,

$$F + R_0 = E_1 + R_1 = M_1$$

Solute balance gives,

$$F y_F + R_0 x_0 = E_1 y_1 + R_1 x_1$$

Solvent balance gives

$$F(1 - y_F) + R_0(1 - x_0) = E_1(1 - y_1) + R_1(1 - x_1)$$

When the solids and the solvent are mixed together in a stage (say, stage 1), the effective value of 'N', called N_{M_1} , will be given by

$$N_{M_1} = \frac{B}{F + R_0} = \frac{B}{M_1}$$

Similarly the concentration of solute after thorough mixing in the stages is given by,

$$y_{M_1} = \frac{y_F F + R_0 x_0}{F + R_0}$$

Using the values of y_{M_1} , N_{M_1} and N vs. x , y diagram, one can determine the concentration and flow rates of leaving streams as indicated below.

(The co-ordinates (y_{M_1}, N_{M_1}) can be represented as shown in Fig. 11.13 in N vs x , y diagram).

Steps

- (i) Draw the N vs x , y diagram.
- (ii) Draw the distribution curve.
- (iii) Locate F (y_F, N_F) and R_0 (x_0, N_0).
- (iv) Join $R_0 F$.
- (v) Locate M_1 (y_{M_1}, N_{M_1}) in $R_0 F$ line.
- (vi) Draw the tie line $R_1 E_1$ passing through M_1 with the help of distribution curve and read N_1 from N vs y curve.
- (vii) $E_1 = B/N_1$ (weight of solution associated with sludge)

We know that,

$$F + R_0 = E_1 + R_1$$

Hence, R_1 , the weight of clear solution can be estimated.

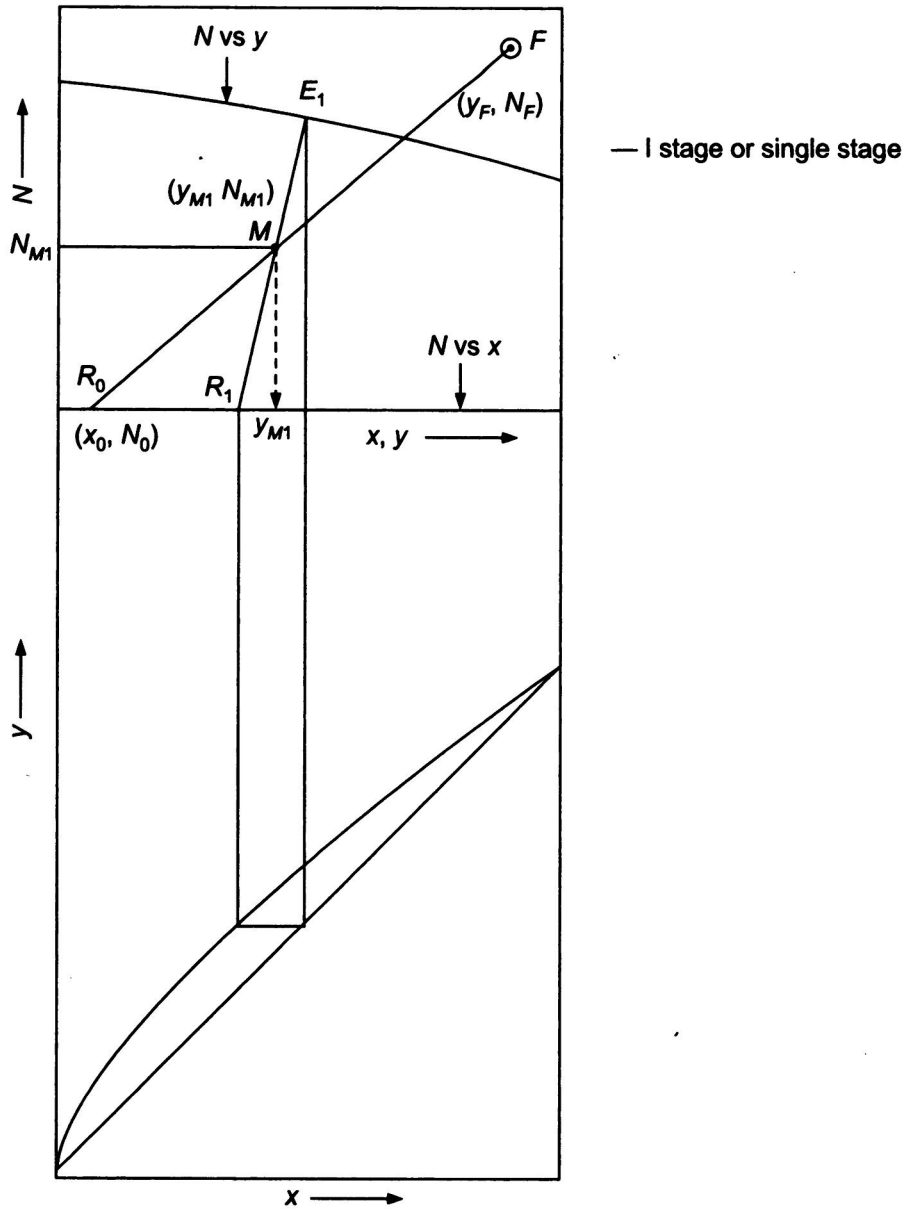


Fig. 11.13 Single stage operation.

11.7 MULTISTAGE CROSS-CURRENT LEACHING

In a multistage cross-current leaching E_1 stream from the I stage becomes the feed stream for the II stage and the E_2 stream from the II stage becomes the feed stream for the III stage. In each stage the mixture is contacted with a fresh stream of solvent.

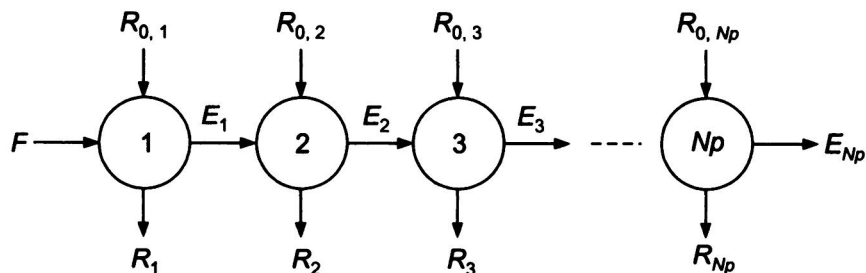


Fig. 11.14 A typical multistage cross-current operation.

Steps

- (i) Proceed as per the procedure mentioned in steps (i) to (vii) of single stage operation of section 11.6.
- (ii) Join E_1 with R_0 and locate M_2 (y_{M_2}, N_{M_2}). Generally $R_{01}, R_{02}, R_{03}, \dots, R_{0, NP}$ are all same $R_{01} = R_{02} = R_{03} \dots = R_{0, NP} = R_0$.
- (iii) Draw the tie line E_2R_2 passing through M_2 and locate N_2 .
- (iv) $E_2 = \frac{B}{N_2}$
- (v) We know from material balance, $E_1 + R_0 = E_2 + R_2$.
- (vi) Hence, the unknown quantity R_2 (weight of clear solution) can be determined since the remaining quantities (E_1, R_0 and E_2) are all known.
- (vii) Proceed in the same manner for other stages also.

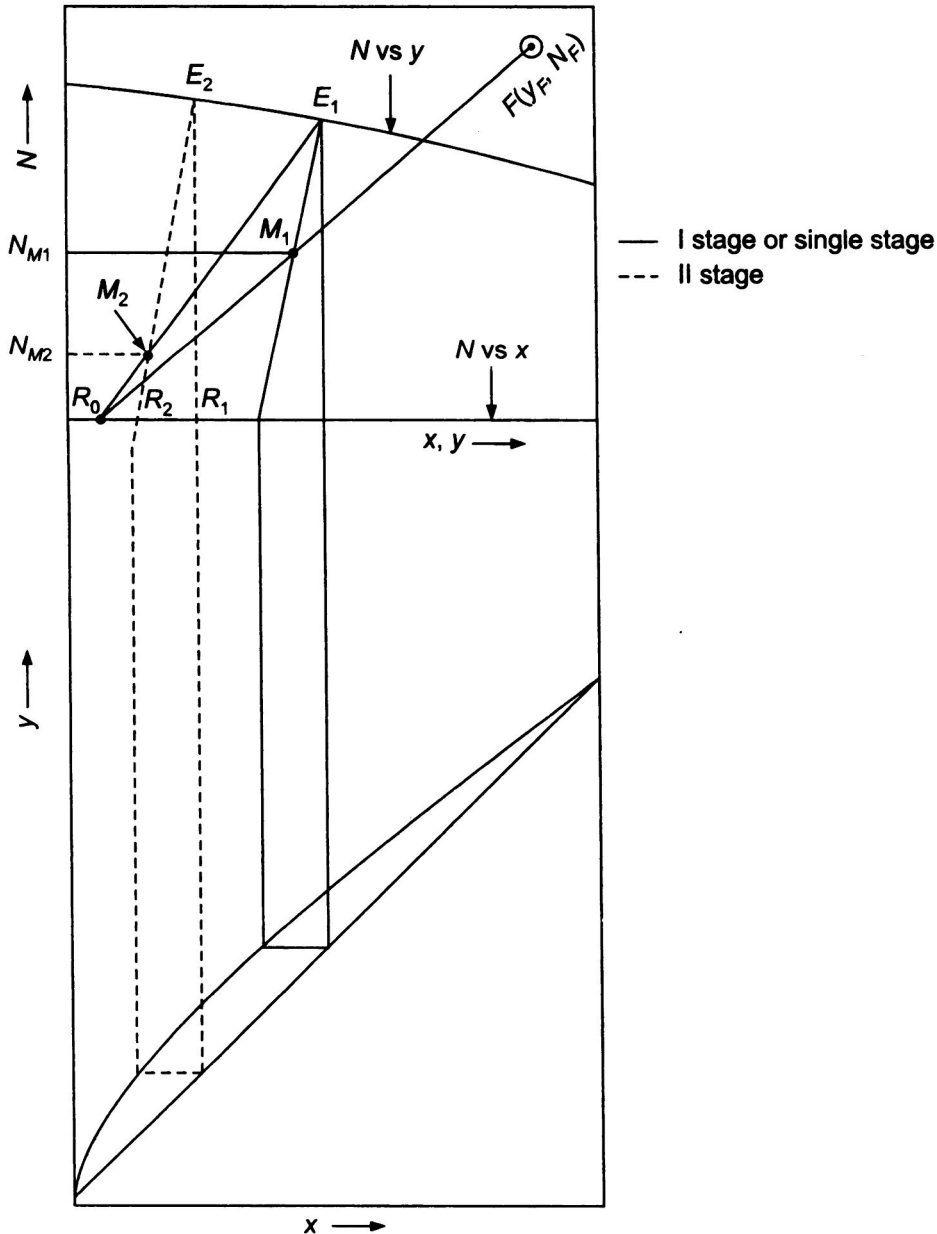


Fig. 11.15 Multistage cross-current operation.

11.8 MULTISTAGE COUNTERCURRENT OPERATION

Solution balance for the system as a whole gives,

$$F + R_{N_p+1} = R_1 + E_{N_p} = M$$

where M is the total mass of B (inert) free mixture.

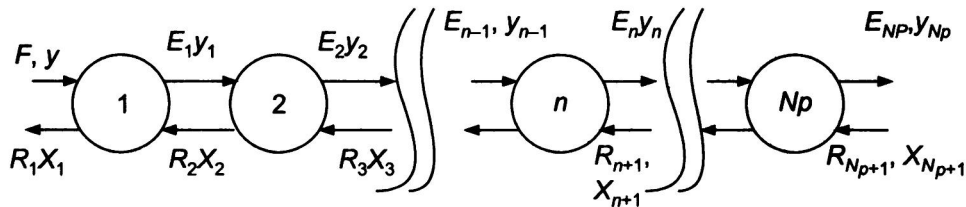


Fig. 11.16 A typical multistage countercurrent operation.

Solute balance gives,

$$F \cdot y_F + R_{N_p+1} \cdot X_{N_p+1} = R_1 x_1 + E_{N_p} \cdot y_{N_p} = M \cdot y_M$$

where,

$$N_M = \frac{B}{F + R_{N_p+1}}$$

$$y_M = \frac{F y_F + R_{N_p+1} X_{N_p+1}}{F + R_{N_p+1}}$$

$$F - R_1 = E_{N_p} - R_{N_p+1} = \Delta_R$$

A solution balance for the first two stages gives

$$F + R_2 = R_1 + E_1$$

i.e.

$$F - R_1 = E_1 - R_2 = \Delta_R$$

Similarly a solution balance for the first two stages yields

$$F - R_1 = E_2 - R_3 = \Delta_R$$

It clearly indicates that the difference in flow between streams at either ends in each stage remains constant.

In a typical operation, the number of stages (for a given recovery and a given amount of solvent) or concentration of solute in the leaving stream (for a given number of stages and solvent used) or the amount of solvent (for a given number of stages and percentage recovery) will be needed.

11.8.1 Analysis of Variable Underflow System

11.8.1.1 Case I

Determination of stages for a specified recovery or final concentration:

Steps

1. Draw N vs x and N vs y and the distribution curve.
2. Locate the points F , E_{N_p} and R_{N_p+1} .

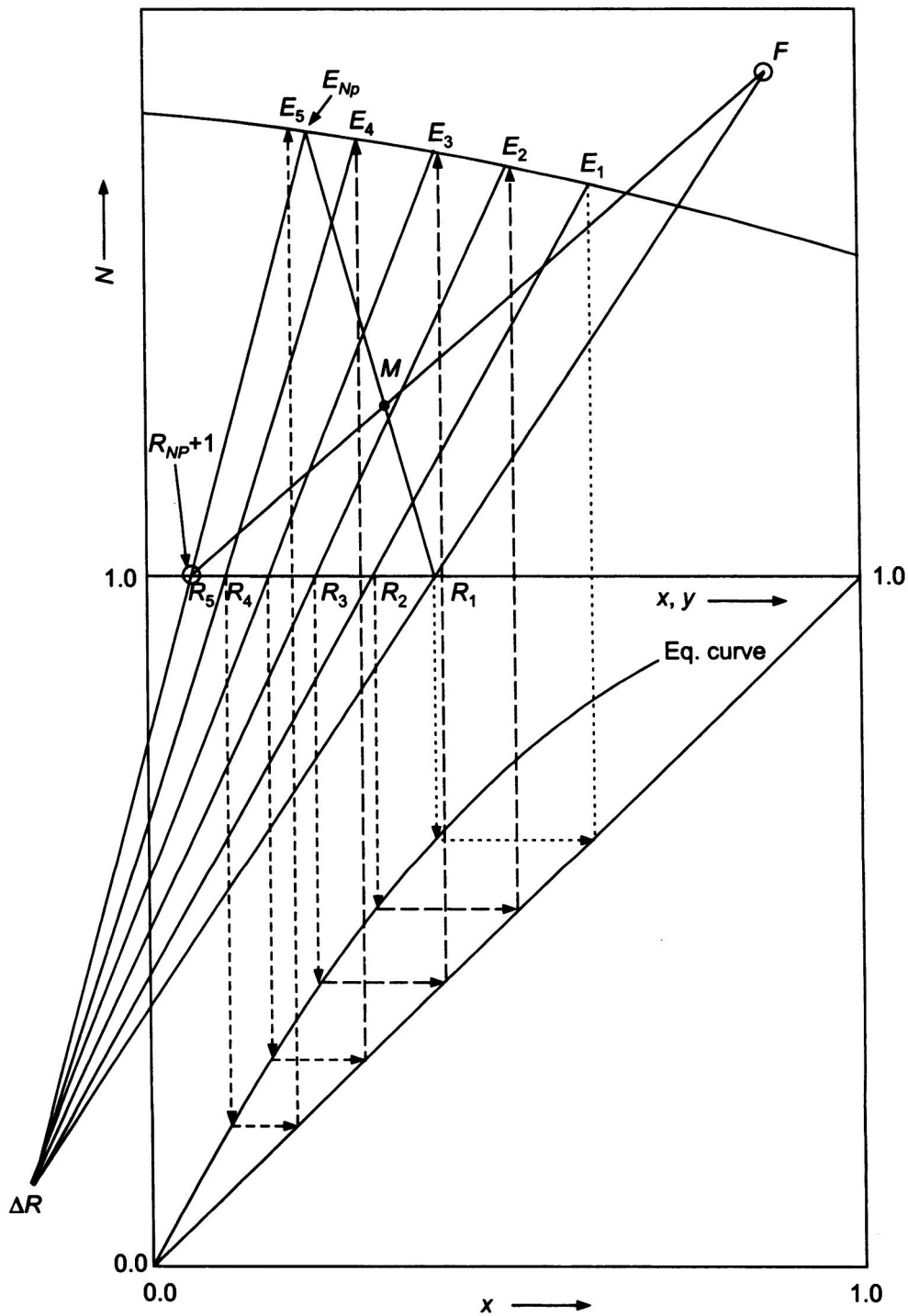


Fig. 11.17 Multistage countercurrent operation.

3. Estimate M (y_M, N_M) and locate it on FR_{N_p+1} line.
4. Join E_{N_p} with M and extend it to cut N vs x curve at R_1 .
5. Join E_{N_p} with R_{N_p+1} and extend it.
6. Join F with R_1 and extend it to cut the $E_{N_p}R_{N_p+1}$ line and call the point of intersection as Δ_R .
7. Using R_1 and equilibrium curve, locate E_1 . This corresponds to stage 1.
8. Join E_1 with Δ_R and this line cuts N vs x curve at R_2 .

9. Using R_2 and equilibrium curve, locate E_2 . This corresponds to stage 2.
10. Proceed in this manner till E_{N_p} is reached or crossed.
11. From this, the number of stages N_p can be determined.

11.8.1.2 Case II

If final concentration is needed or percentage recovery is needed for a given number of stages:

Assume some E_{N_p} value and proceed as mentioned above and verify whether the assumed E_{N_p} matches the given number of stages. If it does not match, assume a new value for E_{N_p} and proceed till the given number of stages and the assumed E_{N_p} value match.

11.8.1.3 Case III

If the solvent amount is needed:

Assume some solvent flow rate and check whether the E_{N_p} and stages match. If they do not match, assume a different value and proceed till the assumed E_{N_p} value and the given stages match.

11.8.1.4 Case IV

Minimum solvent requirement:

It is a specific solvent quantity at which the operating line becomes a tie line, i.e. FR_1 or E_1R_2 or E_2R_3 , ..., becomes a tie line.

11.8.2 Number of Stages for a Constant Underflow System

The number of stages can be determined easily for constant underflow systems as the slope is constant ($m = y/x$) and operating line is straight, by using the Kremser, Brown and Souder's equation.

$$\frac{y_F - y_{N_p}}{y_1 - mx_{N_p+1}} = \frac{\left(\frac{R}{mE}\right)^{N_p+1} - \left(\frac{R}{mE}\right)}{\left(\frac{R}{mE}\right)^{N_p+1} - 1}$$

WORKED EXAMPLES

1. Oil is to be extracted from halibut liver in a countercurrent extraction battery. The entrainment of solution by the granulated liver mass is given below.

kg solution retained/ kg of exhausted liver	0.035	0.042	0.05	0.058	0.068	0.081	0.099	0.12
kg of oil/ kg of solution	0	0.1	0.2	0.3	0.4	0.5	0.6	0.68

In the extraction battery change is to be 100 kg based on completely exhausted liver. The unextracted liver contains 0.043 kg of oil/kg of exhausted material. 95% recovery is desired. The final extract is to contain 0.65 kg oil/kg of extract. The ether used as solvent is free from oil. How many kg of ether is needed per kg of liver? How many extractors are needed?

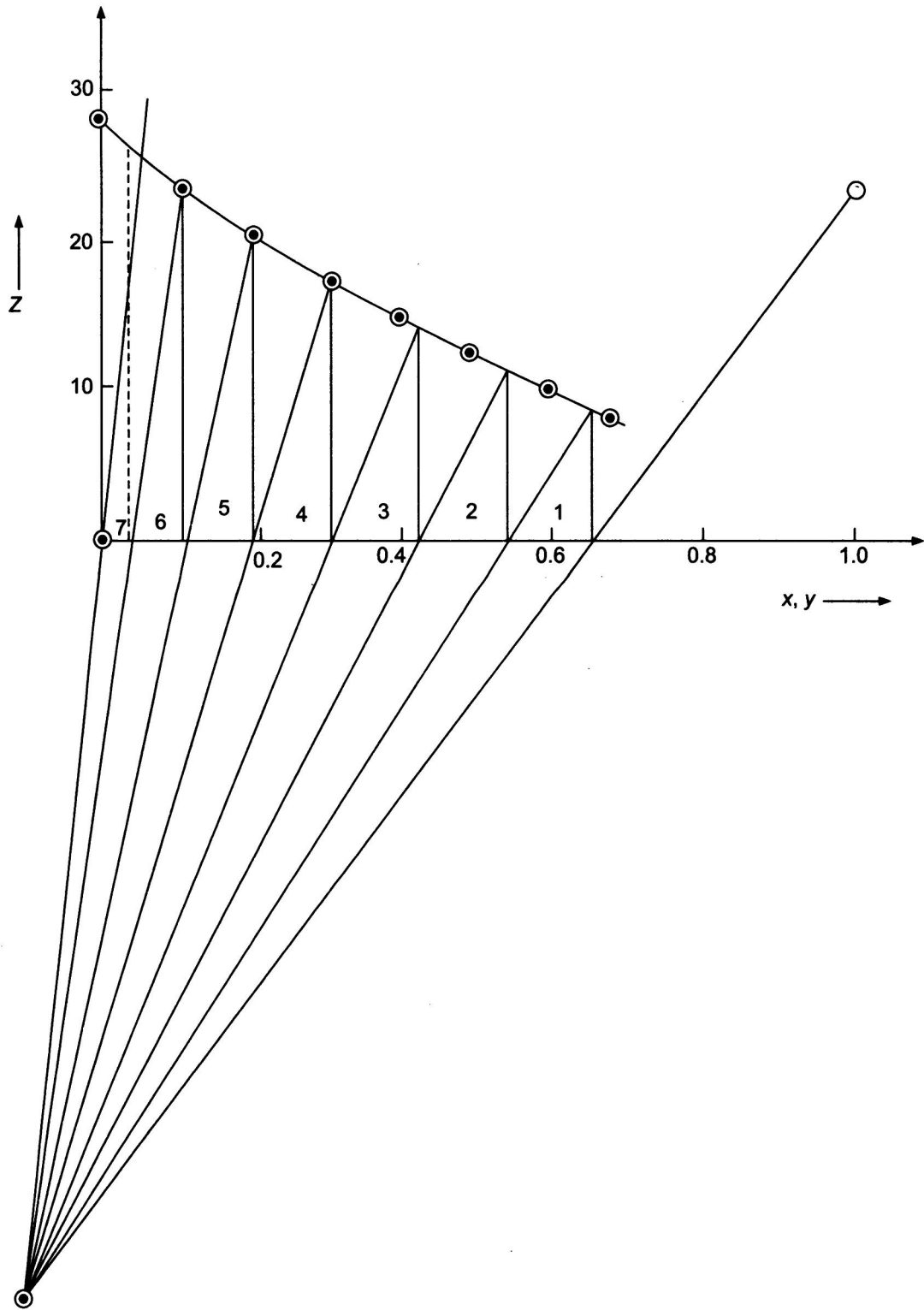


Fig. 11.18 Example 1.

Solution.

$\frac{\text{kg solution retained}}{\text{kg exhausted liver}}, 1/N$	0.035	0.042	0.05	0.058	0.068	0.081	0.099	0.12
$\frac{\text{kg oil}}{\text{kg solution}}, x, y$	0	0.1	0.2	0.3	0.4	0.5	0.6	0.68
$N = \frac{\text{kg exhausted liver}}{\text{kg solution retained}}$	28.6	23.8	20.0	17.25	14.7	12.35	10.1	8.3

Basis: 100 kg of exhausted liver

i.e.

$$B = 100 \text{ kg}$$

$$C \text{ (oil)} = 100 \times 0.043 = 4.3 \text{ kg}$$

$$F = A + C$$

$$A = 0 \text{ (solvent is not present)}$$

$$N_F = \frac{B}{A + C} = \frac{100}{4.3} = 23.26$$

$$y_F = \frac{C}{A + C} = \frac{4.3}{0 + 4.3} = 1.0$$

\therefore Feed point F is given by $(N_F, y_F) = (23.26, 1.0)$

The final extract contains 0.65 kg of oil/kg of extract

$\therefore R_1$ is given by $(N_1, x_1) = (0, 0.65)$

R_{N_p+1} is given by $(N_{N_p+1}, x_{N_p+1}) = (0, 0)$

95% recovery of oil is to be achieved.

\therefore 5% oil leaves with the liver.

i.e. oil leaving is $4.3 \times 0.05 = 0.215 \text{ kg}$

$$\therefore y_{N_p} = \frac{0.215}{A + 0.215}$$

$\therefore E_{N_p}$ is given by (N_{N_p}, y_{N_p})

$$N_{N_p} = \frac{B}{A + C} = \frac{100}{A + 0.215}$$

$$\therefore \text{Slope of operating line} \left(\frac{B}{A + C} \right) / \left(\frac{C}{A + C} \right) = \frac{100}{0.215} = 465$$

From the plot $E_{N_p} = (25.5, 0.055)$

Stages needed = 7.

$$N_{N_p} = 25.5 = \frac{B}{A + C}$$

$$\therefore A + C = \frac{B}{25.5} = \frac{100}{25.5} = 3.92$$

$$y_{N_p} = 0.055 = \frac{C}{A + C}$$

$$A + C = 3.92$$

$$\therefore A = 3.92 - C = 3.92 - 0.215 = 3.705 \text{ kg}$$

i.e. amount of solvent in liver = 3.705 kg

Quantity of ether used

Extract contains 0.65 kg oil/kg extract

i.e. Extract contains 0.35 kg ether/kg extract

$$R_1 = \frac{C}{A + C} = 0.65$$

But C, Oil in extract = Total oil fed – Oil in exhausted liver
 = 4.3 – 0.215 = 4.085 kg

$$\therefore R_1 = 0.65 = \frac{4.085}{A + 4.085}$$

$$\therefore A = 2.2 \text{ kg}$$

$$\therefore \text{Total ether used} = \text{Amount in extract} + \text{Amount in exhausted liver} \\ = 2.2 + 3.705 = 5.905 \text{ kg.}$$

2. 10 tonnes/hour of dry seashore sand containing 1% by weight of salt is to be washed with 10 tonnes/hour of fresh water running countercurrent to the sand through two classifiers in series. Assume perfect mixing of sand and water occurs in each classifier and that the sand discharged from each classifier contain one part of water for every two parts of sand by weight. If the washed sand is dried in kiln, what % of salt will it retain? What wash rate is required in a single classifier in order to wash the sand equally well?

Solution.

Let x be the fraction of salt in the underflow discharge from stage 1.

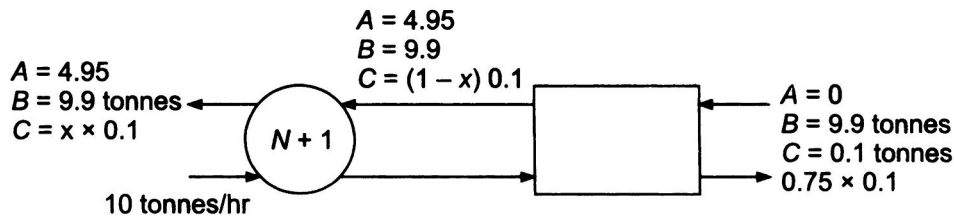


Fig. 11.19 Example 2.

Sand entering 9.9 tonnes/hour. Salt entering = 0.1 tonnes/hour. 1 part of sand discharged associated with 0.5 parts of water.

9.9 tonnes of sand leaving will be associated with $9.9/2 = 4.95$ tonnes of water each stage.

By Coulson–Richardson method,

$$\frac{S_{n+1}}{S_1} = \frac{R - 1}{R^{n+1} - 1}$$

where S_1 = Quantity of solute in the sludge coming out from stage 1,

S_{n+1} = Quantity of solute in the sludge coming out from stage $n + 1$.

$$R = \frac{\text{Quantity of solution in overflow (solute or solvent)}}{\text{Quantity of solution in underflow (solute or solvent)}}$$

$$R = \frac{\text{weight of solution in overflow}}{\text{weight of soluton in underflow}} = \frac{10}{4.95} = 2.02$$

$$S_{n+1} = (x) \times (0.1)$$

$$S_1 = (1 - x) \times (0.1)$$

$$\therefore = \frac{2.02 - 1}{(2.02)^2 - 1} = \frac{x}{(1 - x)} = \frac{1.02}{3.08}$$

$$\therefore 3.02 = \frac{1 - x}{x}$$

$$\therefore x = 0.249$$

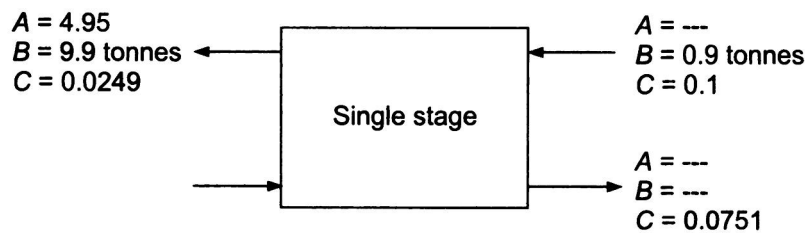


Fig. 11.20 Example 3.

Concentration in underflow

$$\therefore x_1 = \frac{C}{A + C} = \frac{0.0249}{4.95 + 0.0249} = 0.5 \times 10^{-2}$$

$$x_1 \text{ in overflow (same as underflow)} = \frac{0.0751}{A + 0.0751} = 0.5 \times 10^{-2}$$

$\therefore A = 14.93$ (amount of water with extract)

Amount of water with sand = 4.95

$$\therefore \text{Total feed water} = \text{Water in extract} + \text{Water in sand} = 14.93 + 4.95 = 19.88$$

3. 100 tonnes of underflow feed containing 20 tonnes of solute. 2 tonnes of H₂O, 78 tonnes of inerts are to be leached with water to give an overflow of concentration, 15% solute. 95% recovery is desired. The underflow from each stage carries 0.5 kg of solution/kg of inert. Estimate the number of stages needed.

Solution.

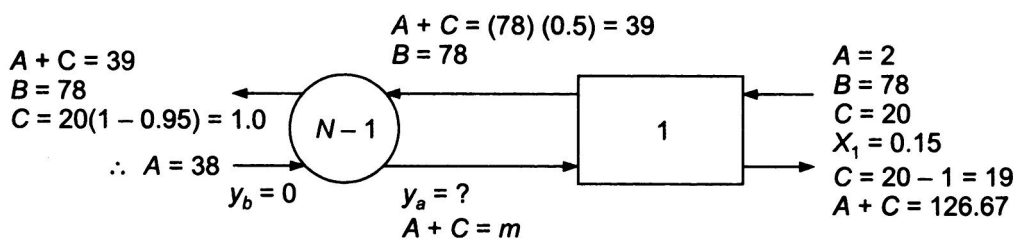


Fig. 11.21 Example 3.

$$y_b^* = \frac{C}{A + C} = \frac{1}{39} = 0.0256$$

x_1 (Desired outlet concentration of overflow) = 0.15

i.e.
$$\frac{C}{A + C} = 0.15 = \frac{19}{A + 19}$$

$\therefore A + C = 126.67$ tonnes

Let us make a mass balance around stage 1.

Entering liquid = Leaving liquid

$$22 + m = 126.67 + 39$$

$\therefore m = 143.67$ tonnes

Similarly making a solute balance, we get

$$20 + 143.67 y_a = 19 + 39 \times 0.15$$

$\therefore y_a = 0.034$

Solving by McCabe's method, we get

$$y_b = 0; y_b^* = 0.0256; y_a = 0.034; y_a^* = 0.15$$

(\because the leaving streams are in equilibrium)

$$(N - 1) = \frac{\log \left[\frac{y_b - y_b^*}{y_a - y_a^*} \right]}{\log \left[\frac{y_b - y_a}{y_b^* - y_a^*} \right]} = \frac{-0.6562}{-0.5633} = 1.165$$

$$N = 2.165$$

Baker's method:

$$\frac{R^{n+1} - 1}{R - 1} = \frac{S_1}{S_{n+1}}$$

$$S_1 = 39 \times 0.15 = 5.85; S_{n+1} = 1.0$$

$$R = \frac{\text{solution/solute or solvent in overflow}}{\text{solution/solute or solvent in underflow}} = \frac{143.7}{39} = 3.685$$

$$\frac{3.685^{n+1} - 1}{2.685} = \frac{5.85}{1}; n + 1 = 2.159 \text{ stages}$$

4. A plant produces 100 tonnes/day of TiO_2 pigment which must be 99.9% pure when dried. The pigment produces by precipitation and the material as prepared is contaminated with 1 ton of salt solution containing 0.55 ton of salt/ton of pigment. The material is washed countercurrently with water in a number of thickeners arranged in series. How many thickeners will be required if water is added at the rate of 200 tonnes/day and the solid discharged from each thickener removed 0.5 ton of solvent/ton of pigment.

What will be the number of thickeners if the amount of solution removed in association with pigment varies in the following way with the concentration of the solution in the thickeners.

x	0	0.1	0.2	0.3	0.4	0.5
N	3.333	3.125	2.94	2.78	2.63	2.5

Solution.

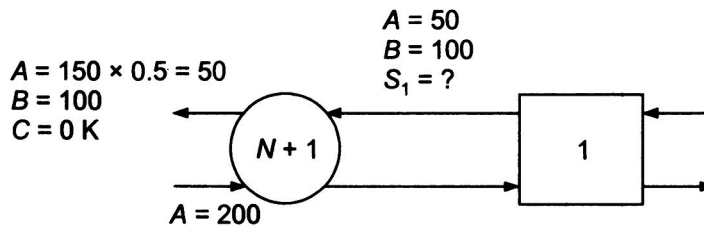


Fig. 11.22 Example 4.

Concentrated wash liquor is fed with the feed top concentrator = 1

$$A + C = 100; A = 45; B = 100$$

$$C = 0.55 \times 100 = 55$$

$$N_F = \frac{B}{A + C} = 1.0$$

$$y_F = \frac{C}{A + C} = 0.55$$

$$C = 55 - 0.1 = 54.9$$

$$A = 200 + 45 - 50 = 195$$

$$y = \frac{C}{A + C} = \frac{54.9}{249.9} = 0.22$$

$$x_1 = 0.22 = \frac{C}{A + C}$$

$$\frac{R^{n+1} - 1}{R - 1} = \frac{S_1}{S_{n+1}}$$

$$R = \frac{200}{50} = 4$$

$$S_1 = 14.1$$

$$S_{n+1} = 0.1$$

$$\frac{4^{n+1} - 1}{4 - 1} = \frac{14.1}{0.1}$$

$$\therefore n + 1 = 4.36$$

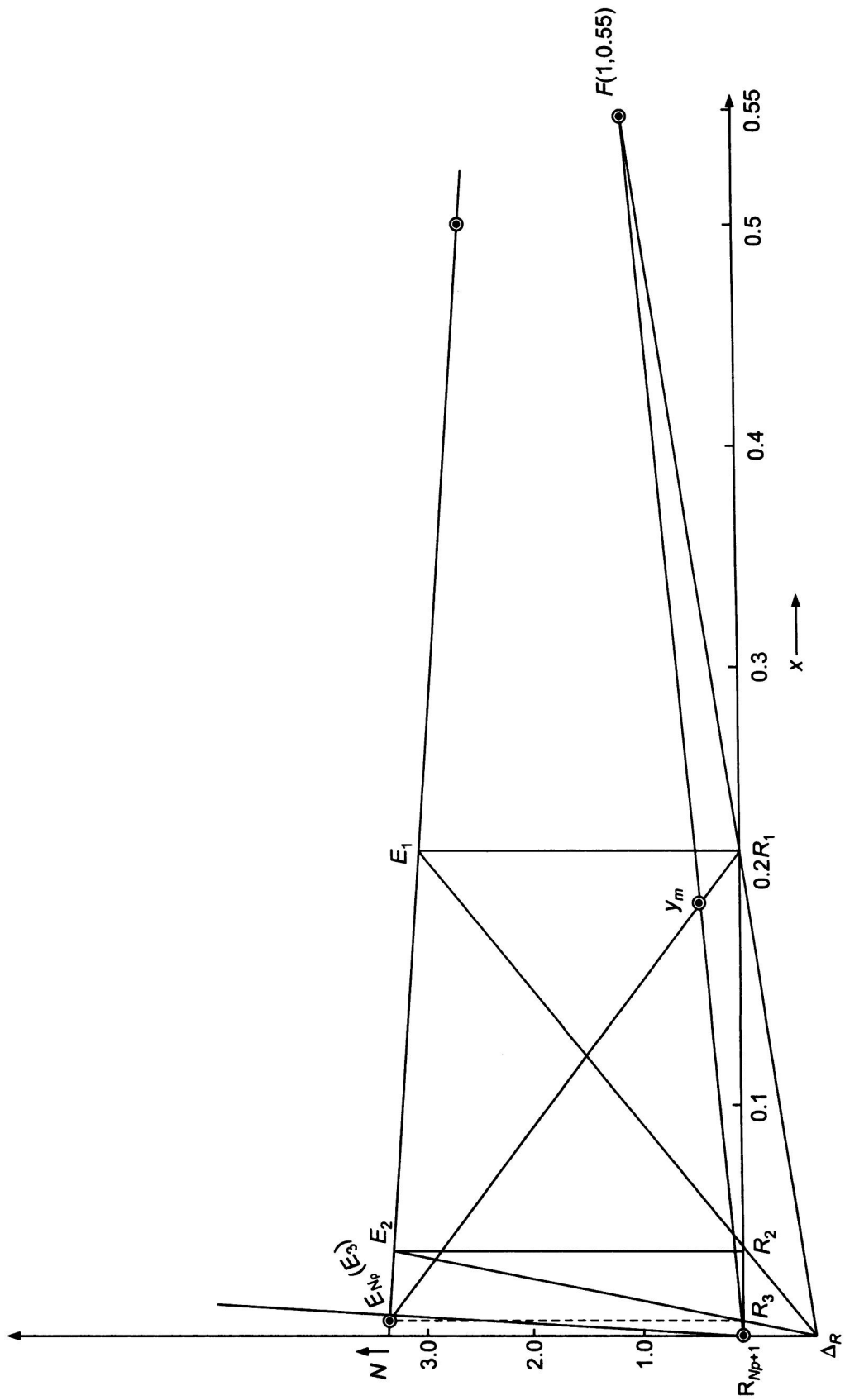


Fig. 11.23 Example 4.

(ii) Feed point F , $(N_F, y_F) = (1, 0.55)$

Leached solids leaving, $E_{N_p} (N_{N_p}, y_{N_p}) = (?, ?)$

Solvent entering, $R_{N_{p+1}} (N_{p+1}, x_{N_{p+1}}) = (0, 0)$

Solution leaving, $R_1 (N_1, x_1) = (0, ?)$

$$\bar{y} = \frac{F \cdot y_F + R_{N_p} x_{N_{p+1}}}{F + R_{N_{p+1}}} = \frac{(100)(0.55) + 0}{100 + 200} = 0.1833$$

$$\bar{N} = \frac{B}{A + C} = \frac{100}{300} = 0.333$$

Join F and $R_{N_{p+1}}$ Locate $\bar{m} (\bar{N}, \bar{y})$.

By stagewise construction, the stages are estimated to be: 4

5. By extraction with kerosene two tonnes of waxed paper per day is to be dewaxed in a continuous countercurrent extraction system. The waxed paper contains 25% paraffin wax by weight and 75% paper pulp. The pulp which retains the unextracted wax must not contain over 0.2 kg of wax/100 kg of wax free pulp. The kerosene used for extraction contains 0.05 kg of wax/100 kg wax free kerosene, experiments show that pulp retains 2 kg of kerosene per kg of wax free pulp. The extract from battery contains 5 kg of wax/100 kg of wax free kerosene. How many stages are needed?

Solution.

Basis: 100 kg of wax and kerosene free pulp

$$\text{Wax in the pulp} = 100 \times \frac{25}{75} = 33.33 \text{ kg}$$

Wax in the solvent = 0.0005 kg of wax/kg of kerosene

Let s be the weight of solvent used.

$$\therefore \text{Total wax entering} = \text{wax from pulp} + \text{wax from kerosene} = 33.33 + 0.0005s$$

$$\text{Wax in the exiting pulp} = 100 \times 0.002 = 0.2 \text{ kg}$$

Wax in the solution leaving

[Solvent entering – solvent carried away in leaving pulp] [Weight ratio of wax to solvent in leaving solution]

$$= [s - (2)(100)] [0.05] = (0.05s - 10) \text{ kg}$$

$$\therefore \text{Total wax output} = (0.05s - 10) + (0.2) = (0.05s - 9.8)$$

Wax input = wax output

$$\text{i.e. } 33.33 + 0.0005s = 0.05s - 9.8$$

$$s = 871.3 \text{ kg}$$

Kerosene in the exhausted pulp = $2 \times 100 = 200$

Kerosene in the extract (overflow) solution = $871.3 - 200 = 671.3 \text{ kg}$

$$\text{i.e. wax in the extract (overflow) solution} = 671.3 \times \frac{0.5}{100} = 33.565 \text{ kg}$$

Concentration in underflow in II unit = Concentration in overflow from I stage

Wax in underflow leaving I solution = Weight of kerosene in underflow × wax concentration

$$= (200) \left(\frac{5}{100} \right) = 10 \text{ kg}$$

The wax in the overflow from II cell to I cell by wax balance

[Wax in underflow leaving I + wax in overflow solution leaving I – wax in pulp entering I]

$$10 + 33.565 - 33.33 = 10.235 \text{ kg}$$

Concentration of this solution is $\frac{10.235}{871.3} = 0.0117$

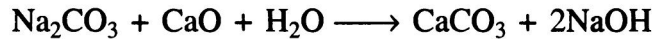
$$x_a = y_a^* = 0.05 \text{ and } y_a = 0.0117$$

$$x_b = y_b^* = \frac{0.2}{200} = 0.001, \text{ } y_b = 0.0005$$

$$N - 1 = \frac{\log \left[\frac{0.0005 - 0.001}{0.0117 - 0.05} \right]}{\log \left[\frac{0.0005 - 0.0117}{0.001 - 0.05} \right]} = \frac{-1.88423}{-0.641} = 2.94$$

∴ $N = 3.94$ stage; $N \approx 4$ stage

6. A five-stage countercurrent extraction battery is to be used to extract the sludge from the reaction



The CaCO_3 leaving each carries with it 1.5 times its weight the solution, in flowing from one unit to other. It is desired to recover 98% of NaOH. The products from the reaction enter the first unit with no excess reactant but with 6.5 kg of water/kg of CaCO_3 .

- (i) How much wastewater must be used for 1 kg of CaCO_3 ?
- (ii) What is the concentration of leaving solution assuming CaCO_3 is insoluble?
- (iii) Using the same quantity of wastewater, how many units must be employed to recover 99.5% of NaOH.

Solution.

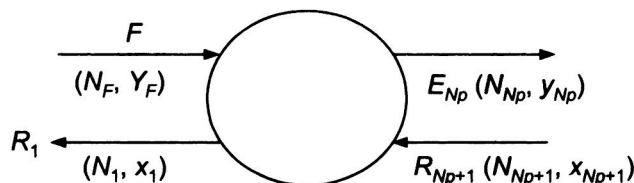


Fig. 11.24 Example 6.

Basis:

100 kg CaCO₃ formed

B (Inert) : 100 kg

A (Solvent) : 650 kg

C (Solute) : 80 kg (from stoichiometry)

$$\therefore N_F = \frac{B}{A + C} = \frac{100}{650 + 80} = 0.137$$

$$y_F = \frac{C}{A + C} = \frac{80}{730} = 0.1096$$

$$\therefore F (0.137, 0.1096)$$

$$N_{N_p} = \frac{1}{1.5} = 0.667$$

Recovery of NaOH is 987 = 78.4 kg

\therefore NaOH in leaving stream = 1.6 kg

$$\therefore y_{N_p} = \frac{C}{A + C} = \frac{C}{E_{N_p}}$$

$$E_{N_p} = \frac{B}{N_{N_p}} = \frac{100}{0.667} = 150$$

$$\therefore y_{N_p} = \frac{1.6}{150} = 0.0107$$

Point E_{N_p} is $(N_{N_p}, y_{N_p}) = (0.667, 0.0107)$

Assume x_1 and hence locate R_1 . $(0, x_1)$ locate E_{N_p} $(0.667, 0.0107)$, F $(0.137, 0.1096)$ and R_{N_p+1} $(0, 0)$

Join E_{N_p} , R_{N_p+1} and F , R_1 and produce them to cut at Δ_R .

By stepwise construction check whether both five stages and E_{N_p} (assumed) match.

If not, make a fresh assumption of x_1 and proceed till the stages and x_1 match.

By trial and error $x_1 = 0.1$

Total amount of wastewater

(i) Water in sludge $(A + C) =$ Weight of solution in sludge – weight of solute = $150 - 1.6 = 148.4$ kg

(ii) Weight of water in overflow

$$\text{Concentration in overflow} = x_1 = \frac{C}{A + C} = \frac{78.4}{A + C} = 0.1$$

Weight of solution $A + C = 784$ kg

\therefore Weight of solvent $(A) = 784 - 78.4 = 705.6$ kg

\therefore Total weight of water added = $148.4 + 705.6 - 650 = 204$ kg

Concentration of leaving solution from each stage:

$$x_1 = 0.1; x_2 = 0.068; x_3 = 0.044; x_4 = 0.026; x_5 = 0.0107$$

(iii) For 99.5% recovery:

$$\text{Concentration of NaOH leaving} = 0.995 \times 80 = 0.14.$$

$$\frac{F}{R} (N_F, y_F) = F(0.137, 0.1096)$$

$$R_1(N_1, x_1) = R_1(0, 0.1)$$

$$y_{N_p} = \frac{C}{A + C} = \frac{C}{E_{N_p}}$$

$$\therefore E_{N_p} = \frac{B}{N_{N_p}} = 150$$

$$\therefore y_{N_p} = \frac{0.4}{150} = 2.667 \times 10^{-3} = 0.002667$$

$$E_{N_p} (0.667, 2.667 \times 10^{-3})$$

$$R_{N_p+1}(0, 0)$$

By stagewise construction, we find the number of stages as 5.

In the previous problem worked out, it is found that the sludge retains the solution varying with the concentration as follows:

NaOH	0	5	10	15	20
$\frac{\text{kg of solution}}{\text{kg of CaCO}_3}, \frac{1}{N}$	1.5	1.75	2.2	2.7	3.6
N	0.667	0.571	0.455	0.370	0.278

It is desired to produce a 10% solution of NaOH. How many stages must be used to recover 99.5% of NaOH?

Recovered NaOH = 99.5%

i.e. $99.5 \times 80 = 79.6 \text{ kg}$

Solute = 0.4 kg

$$x = \frac{C}{A + C} = \frac{79.6}{A + 79.6} = 0.1$$

$$\therefore A = 716.4 \text{ kg}$$

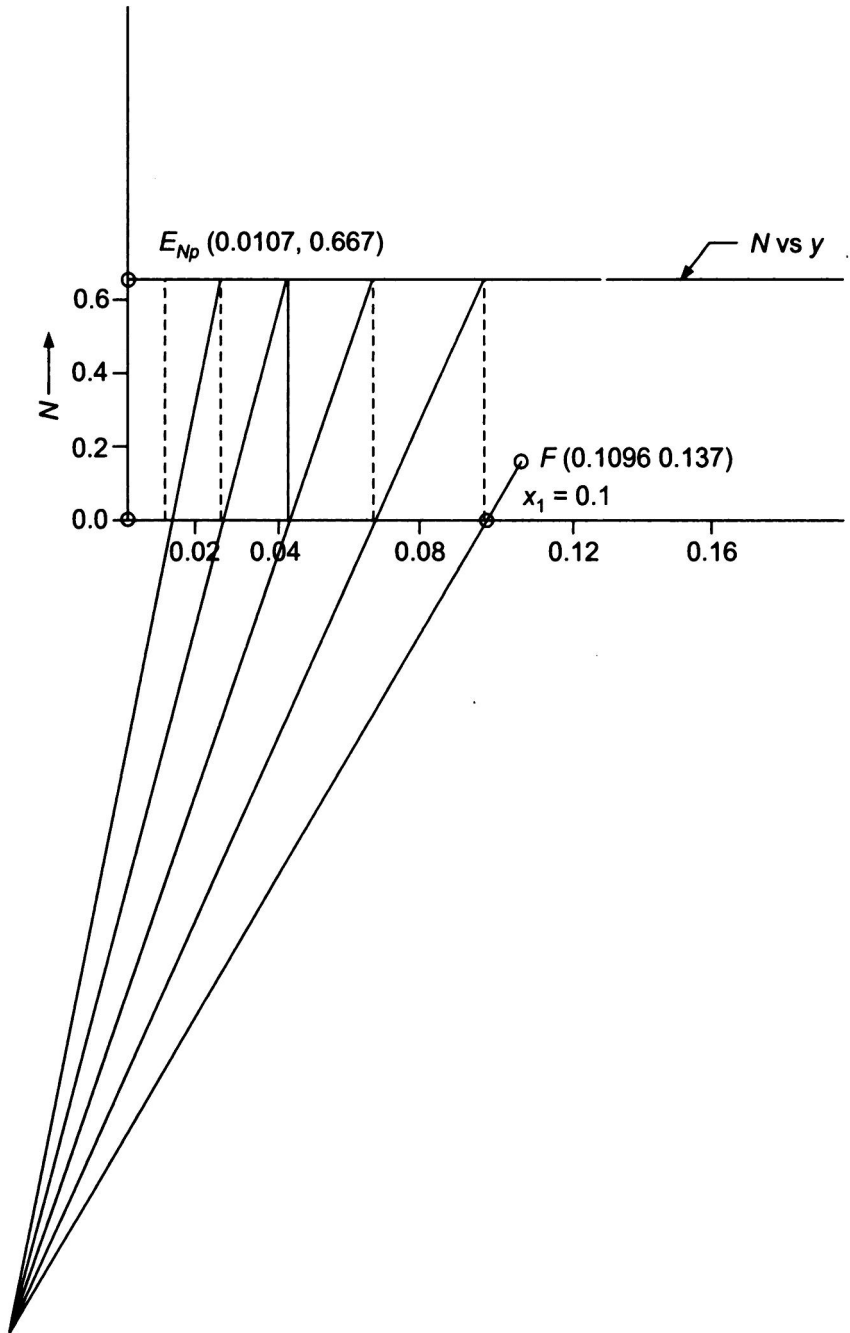


Fig. 11.25 Example 6.

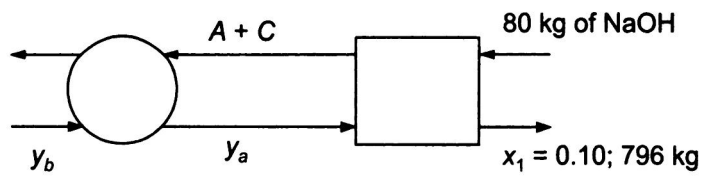


Fig. 11.26 Example 6.