Combined Heat and Mass Transfer

Lec 3: Solid Liquid extraction (Leaching)

Content

Introduction, Principles of leaching, Equilibrium Diagram, Leaching Calculations, Types of Equipments for Leaching

Prof. Zayed Al-Hamamre

Chemical Engineering Department | University of Jordan | Amman 11942, Jordan
Tel. +962 6 535 5000 | 22888

Content

- Introduction
- Principles of leaching
- Equilibrium Phase Diagram for Leaching
- Leaching Calculations
  - Single-Stage Leaching
  - Multistage Cross-current Leaching
  - Multi – Stage Counter Current Leaching
- Types of Equipments for Leaching

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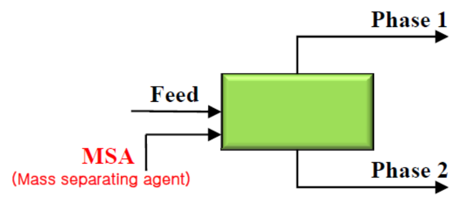
Introduction

➢ Solid liquid extraction (leaching) means the removal of a constituent from a mixture of solids by bringing the solid material into contact with a liquid solvent that dissolves this particular constituent.

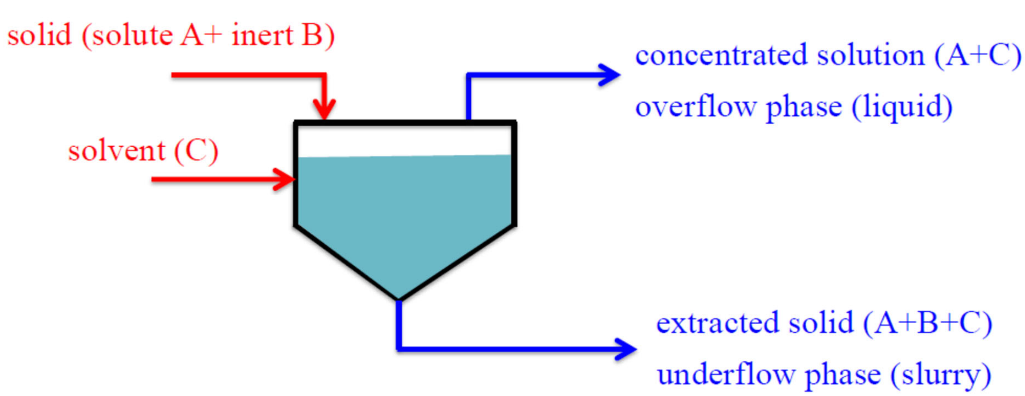


➢ In leaching, to separate the desired solute constituent or remove the undesirable solute component from the solid phase, **the solid is contacted with a liquid phase.**

➢ The two phases are in intimate contact and the solute or solutes can **diffuse from the solid to the liquid phase**, which causes a separation of the components originally in the solid.

➢ The major difference between Leaching and LLE centers about **the difficulty to transport the solid or the solid slurry from stage to stage**.

Introduction



Applications

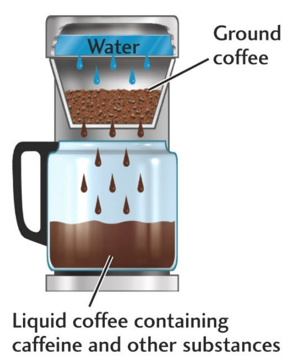
Leaching process for biological substances

- Leaching of soybean oil from flaked soybeans with hexane.
- Leaching of sugar from sugar beets with hot water.
- Production of vegetable oils with organic solvents such as hexane , acetone and ether by extraction the oil peanuts , soybeans, sunflower seeds, cotton seeds and halibut livers.
- Soluble tea is produced by water leaching of tea Leaves.
- In pharmaceutical industry, many different pharmaceutical products are obtained by leaching plan roots, leaves and stems.

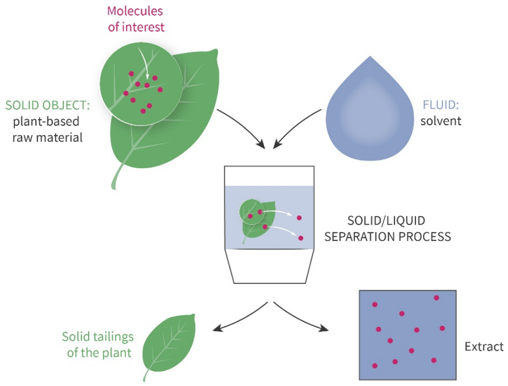
Applications

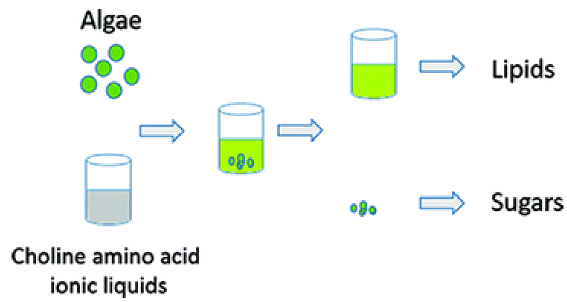
Example:

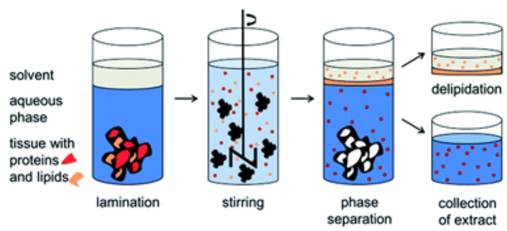
Making coffee from ground coffee beans and tea from tea leaves (The complex mixture of chemicals that give coffee and tea their odor, taste, and physiological effects are leached from the solid by hot water)



Applications







Leaching of mucins (glycosylated proteins)
from homogenized animal material

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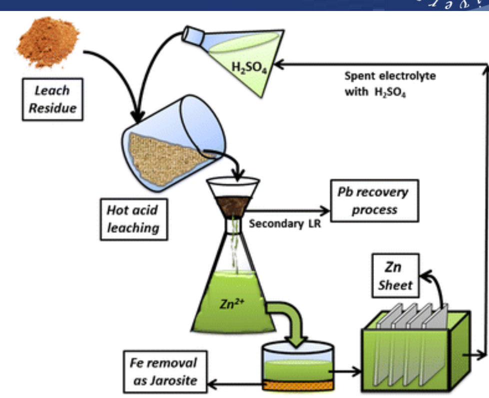
Applications

Metal – processing industries

➢ In metal ores, the desired metal components usually occur with a large amount of undesirable constituents and leaching is used to obtain these metal components in the form of metal salts.

- Gold is leached from its ore using an aqueous sodium cyanide solution.
- Extraction of copper oxide from low grade ores with dilute H_2SO_4 acid.
- Nickel salts are leached using sulfuric acid – ammonia – oxygen mixtures.



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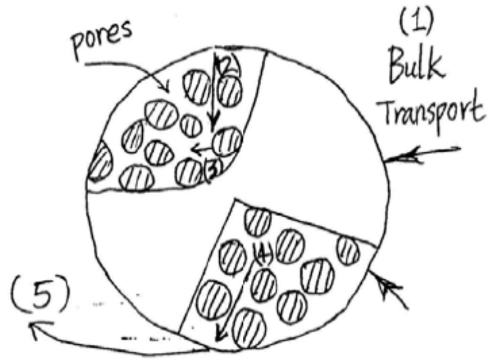
Principles of leaching

- Generally there are **five rate steps** in the leaching process:
  1. The solvent is transferred from the bulk solution to the surface of the solid.
  2. The solvent penetrates or diffuses into the solid (intraparticle diffusion).
  3. The solute dissolves from the solid into the solvent.
  4. The solute diffuses through the mixture to the surface of the solid (intraparticle diffusion).
  5. The solute is transferred to the bulk solution.



Principles of leaching

- The rate of the solvent transfer from the bulk solution to the solid surface (step 1) is quite rapid.
- However, the rate of transfer of the solvent into the solid can be rather slow or rapid.
- This solvent transfer usually occurs initially when the particle are first contacted with the solvent.
- The rate of diffusion of the solute through the solid or solute to the solvent (dissolving step) is often the controlling resistance in the overall leaching process and can depend on a number of different factors.
- If the solid is made of porous the diffusion through the porous solid can be described by an effective diffusivity.
- The resistance to mass transfer to the solute from the solid surface to the bulk solvent is generally quite small compared to the resistance to the diffusion within the solid itself.

Principles of leaching

- **When dissolving step is the controlling process:**
- The rate of mass transfer of the solute A being dissolved to the solution of volume V is:

$$N_A = k_L A(c_{AS} - c_A)$$

Where

N_A is kg mol of A dissolving to the solution/s,

A is the surface area of particles in m^2,

k_L is a mass transfer coefficient in m/s,

c_{AS} is the saturation concentration of the solute A in the solution in kg mol/m^3, and

c_A is the concentration of A in the solution at time t sec in kg mol/ m^3.

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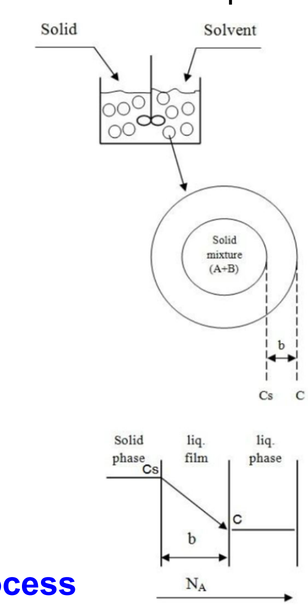
Principles of leaching

- By a material balance, the rate of accumulation of A in the solution is equal to the dissolving flux:

$$\frac{Vdc_A}{dt} = N_A = k_L A(c_{AS} - c_A)$$

Batch System



- Integrating from t = 0 and $c_A = c_{A0}$ to t = t and $c_A = c_A$,

$$\int_{c_{A0}}^{c_A} \frac{dc_A}{(c_{AS} - c_A)} = \frac{k_L A}{V} \int_{t=0}^{t} dt$$

$$\frac{c_{AS} - c_A}{c_{AS} - c_{A0}} = \exp\left(-\frac{k_L A}{V} t\right)$$

This means that, when **dissolving is the controlling process** the solution approaches a saturated condition **exponentially.**

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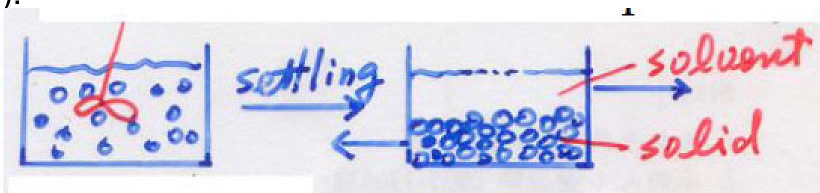
Principles of leaching

<u>Leaching involves two steps which are:-</u>

1- Contacting step: of solvent and the material to be Treated, so as to transfer soluble constituent to the Solvent.

2- Separation step: of the solution formed from the relatively exhausted solids.

- This is a momentum transfer step that can be carried out by (settling or filtration).



- The above two steps may be conducted in separate equipment or in one and the same equipment.
- Solution resulting from separation step is termed **Overflow**, Solids left over are termed **Underflow**.

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Leaching Terminology

<u>1-Contacting Step:</u>

- Basically it's a mass transfer step, it aims at transferring the soluble constituent from the solid phase into the liquid phase by diffusion and dissolution.
- The solute is first dissolved from the surface of the solid, then passes into the main body of the solution by diffusion.
- This process may result in the formation of pores in the solid material which exposes fresh (new) surfaces to subsequent solvent penetration to such surfaces.

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Leaching Terminology

An ideal contacting (mixing) stage yields a product in Thermodynamic Equilibrium→ No mass transfer.

No heat transfer.

No momentum transfer.

Why its difficult to reach an ideal stage behavior, thermodynamic equilibrium ?

- The rate of mass transfer is slow due to the pore resistance: the rate of diffusion of solute out of the pores (capillaries) into the bulk solution are very slow.
- Some part of the contained solute is not exposed to the solvent (inside a non-porous layer or closed pores).

Leaching Terminology

- Sometimes the adsorption of the solute on the surface of the solid is > than the solubility in the solvent, so the realization of an ideal stage needs rather
  - Long times if the operations is carried out batch wise or
  - An excessively large apparatus if the operation is continuous.

How to increase the rate of mass transfer?

1- Increase the agitation speed, thickness of the **boundary layer** decreases.

2- As the temperature increase, 1) the solubility increase, Hence, 2) the diffusion increases , 3) the viscosity decreases leading to 4) decrease in the film thickness, hence 5) the pore resistance decreases.

3-Size reduction of solid to increase the exposed mass transfer area.

Factors Affecting Leaching

- Particle size
  If it is smaller:
  - → Greater specific surface area (+)
  - → Smaller distance within the solid (+)
  - → Lower settling velocity
    - → separation sol./liq. is more difficult(-)
    - → particles can be maintained in solution by agitation (+)
- Solvent Requirements:
  - selectivity
  - low viscosity

Factors Affecting Leaching

- Temperature

  Higher T
  - → higher solubility
  - → D_{AB} increases
  - → lower viscosity

- Agitation of the fluid
  - It increases the turbulence
  - It prevents the settling of solid particles
  - It promotes mass transfer

- Solvent Quantity

- Moisture Content of the Seed

Preparation of solid for leaching

- **Solid preparation depends on:**
  - The proportion of soluble constituent present.
  - Solute distribution throughout the original solid.
  - The original particles size.

- For example, vegetable and animal materials are cellular in structure and the soluble constituents are generally found inside the cells. The wall cell provide another resistance to diffusion.
  - The cell walls of soybean are largely ruptured when the material are reduced in size to about 0.1 mm to 0.5 mm.
  - In leaching of pharmaceutical products from leaves, stems, and roots, drying of the original material rupture the cell walls.

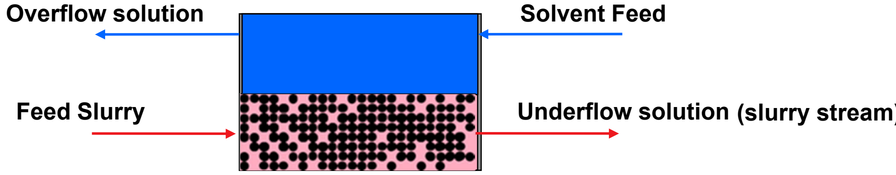
Preparation of solid for leaching

Preparation of Solid Leaching:

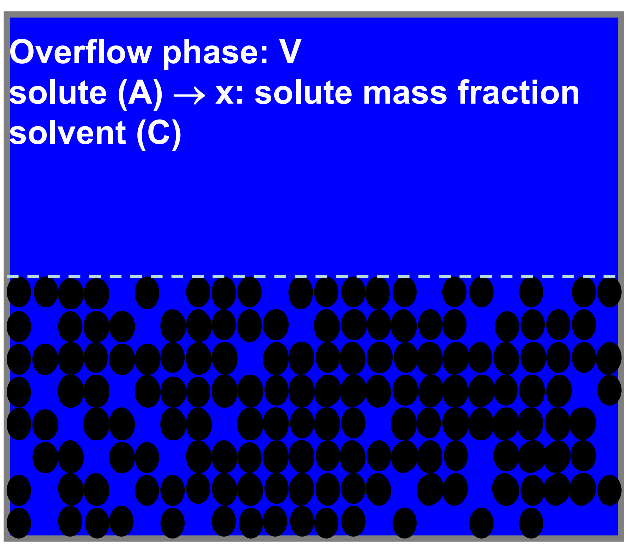
- Crushing and grinding of solid to allow soluble portions are made more accessible to the solvent thus increasing rate of leaching.
- Simple washing can be applied if the soluble substance is widely distributed throughout the whole solid (water can potentially remove water-soluble impurities).
- For biological material, i.e. sugar beet, they are cut into wedge-shape slices for leaching.
  - For soybeans and many vegetable seeds they are ruptures by rolling and flaking in such a way that the vegetable oil is easily accessible to the solvent.
  - For pharmaceutical product e.g. leaves, stems and roots, drying of the material before extraction help to rupture the cell wall and hence solvent can directly dissolved the solute.

Calculation in Leaching

- Single – Stage Leaching
- Multistage Cross-Current Leaching
- Counter – Current Multistage Leaching



Equilibrium Relations in Leaching



Underflow or slurry phase: L
solute (A)
y: solute mass fraction
solvent (C)
Inert or leached solid (B)

At certain temperature and pressure

Equilibrium Relations in Leaching

- We have three component system:
  i. solute (A)
  ii. Inert solid or leached solid (B)
  iii. Solvent (C)

Assumptions:

i. Solute-free solid (inert solid) is insoluble in the solvent.

ii. Solvent present in sufficient amount

iii. Equilibrium is reached when solute is dissolved (sufficient contact time to reach equilibrium)

iv. No adsorption of solute by the solid.

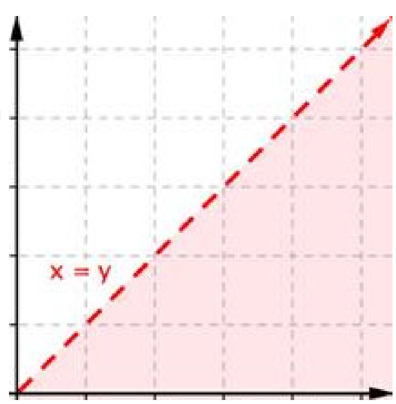
- **UNDERFLOW/SLURRY STREAM** – the settled solid leaving a stage that contains some liquid containing dissolved solid.
- **OVERFLOW STREAM** – Concentration in the liquid solution accompanying the slurry stream.

Equilibrium Phase Diagram for Leaching

→Under the above assumptions: the solute concentration in overflow liquid phase is the same as in the settled slurry "underflow" phase. Hence, on an x-y plot, the equilibrium line is on the 45° line.

y_A

Underflow solution (slurry stream)



x_A

Overflow solution

Equilibrium Phase Diagram for Leaching

- The concentration of inert or insoluble solid B in the slurry mixture is defined as:

$$N = \frac{\text{kg } B}{\text{kg } A + \text{kg } C} = \frac{\text{kg solid}}{\text{kg solution}} = \frac{\text{lb solid}}{\text{lb solution}}$$

- For the overflow phase (no insoluble solid)→$N = 0$
- For the underflow phase, N has dependency upon solute concentration in the liquid.

- The mass compositions of solute A are defined as :

$$x_A \text{ or } x = \frac{\text{kg } A}{\text{kg } A + \text{kg } C} = \frac{\text{kg solute}}{\text{kg solution}} \quad (\text{overflow phase})$$

$$y_A \text{ or } y = \frac{\text{kg } A}{\text{kg } A + \text{kg } C} = \frac{\text{kg solute}}{\text{kg solution}} \quad (\text{slurry or underflow phase})$$

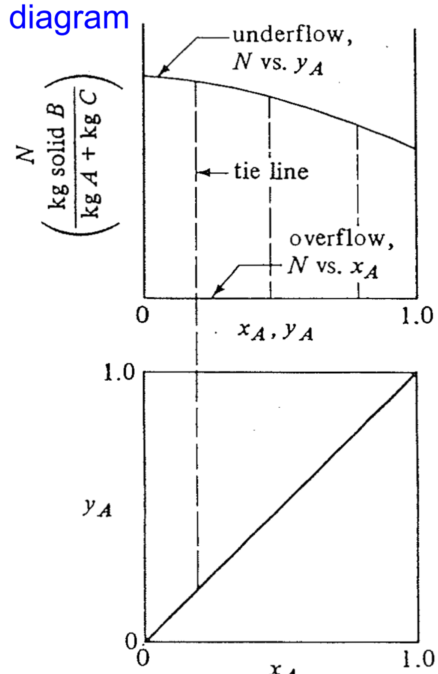
- Remember that at equilibrium: $x_A = y_A$

Equilibrium Phase Diagram for Leaching

Solids-free coordinates:-

- The construction is in general less crowded than in the case of triangular diagram.
- The amounts of various streams should be expressed on solid-free basis.
- The abscissa y (weight fraction of solute on a solid-free basis in the U.F) and x (weight fraction of solute on a solid-free basis in the O.F) is plotted against the amount N (concentration of insoluble solids on the solid-free basis).

N-xy diagram



x-y diagram

Equilibrium Phase Diagram for Leaching

Some notes on the diagram:-

Pure A = 100% , B = 0 , S = 0

$$x \text{ or } y = \frac{A}{A+C} = \frac{A}{A+0} \quad , \quad N = \frac{B}{A+C} = \frac{0}{100+0} = 0$$

Pure C = 100% , A = 0 , B = 0

$$x \text{ or } y = \frac{A}{A+C} = \frac{0}{0+100} = 0 \quad , \quad N = \frac{B}{A+C} = \frac{0}{0+100} = 0$$

Pure B = 100% , A = 0 , S = 0

$$x \text{ or } y = \frac{A}{A+C} = \frac{0}{0+0} = \frac{0}{0} \quad , \quad N = \frac{B}{A+C} = \frac{100}{0+0} = \infty$$

Equilibrium Phase Diagram for Leaching

- For the entering solid feed to be leached, N= N_o is kg inert solid B/kg solute A,

$$N = \frac{\text{kg } B}{\text{kg } A + \cancel{\text{kg } C}} = \frac{\text{kg solid}}{\text{kg solution}} = \frac{\text{lb solid}}{\text{lb solution}}$$

and hence

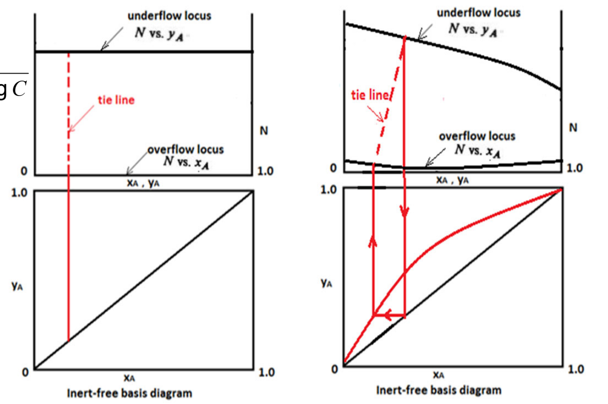
$$y_A \text{ or } y = \frac{\text{kg } A}{\text{kg } A + \cancel{\text{kg } C}} = \frac{\text{kg solute}}{\text{kg solution}} \quad (\text{slurry or underflow phase})$$

y_A = 1.0

Equilibrium Phase Diagram for Leaching

- The shape of the equilibrium curve (settling characteristics) depend on
  - The viscosity and relative density of the liquid in which the solid is suspended, which in turn depends on the concentration of solute in the slurry.
  - The solubility of solute A in solvent C.
  - The adsorptivity of solute A on inert solid B

$$N = \frac{\text{kg } B}{\text{kg } A + \text{kg } C}$$



Typical equilibrium diagrams

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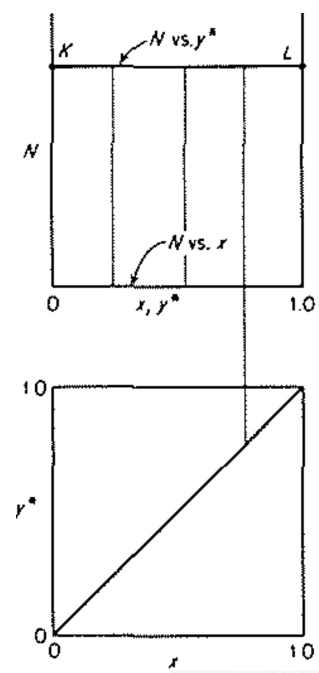
Equilibrium Phase Diagram for Leaching

- If the density and viscosity of the solution do not change with concentration, the mass of the solution retained by the settled solid is constant.
- No adsorption of solute occurs, so that withdrawn solution and solution associated with the solid have the same composition and the tie lines are vertical.
  - This results in an *xy* curve in the lower figure identical with the 45° line, and a distribution coefficient *m,* defined as y^*/x equals unity,
    - Line *KL* is horizontal, indicating that the solids are settled or drained to the same extent at all solute concentrations.
    - It is possible to regulate the operation of continuous thickeners so that this will occur, and the conditions are known as ***constant underflow***.
    - The solution in this case contains no solid B, either dissolved or suspended



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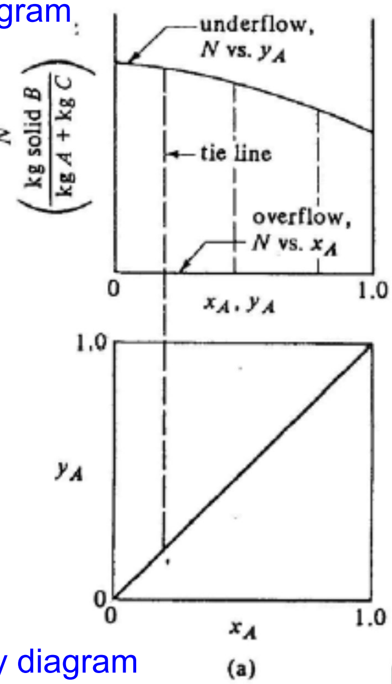
Equilibrium Phase Diagram for Leaching

N-xy diagram

- If the density and viscosity of the solution increase with solute concentration,
  - The rate of solid settling decreases with increasing the solute concentration in the solution
  - More solution will be drawn off with the settled solids and N decrease with increase in x
  - The overflow is represented by the x-axis since it is free of solid
  - The tie lines are vertical as the concentration of solution is uniform throughout.



x-y diagram

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Equilibrium Phase Diagram for Leaching

- For variable underflow conditions with non-uniform solute concentration in the solution

  → y ≠ x (y > x) and the equilibrium diagrams will be as shown

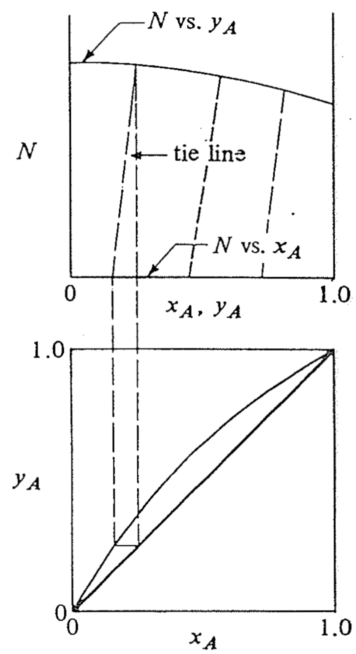
  The tie lines such are not vertical

- This may arise:

  i. The solid B retain some solute adsorbed on the pore surface.

  ii. Some solute remain undissolved due to insufficient contact time

  iii. Solute is soluble in the inert solid B and distributes unequally between liquid and solid phases at equilibrium.



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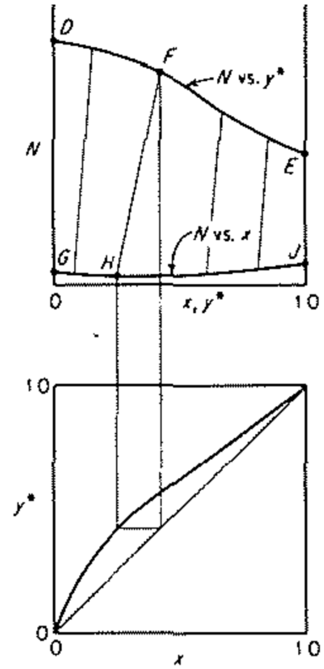
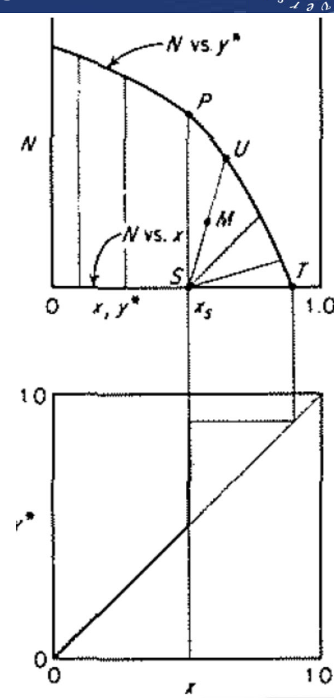
Equilibrium Phase Diagram for Leaching

- Curve *GHJ,* the composition of the withdrawn solution, lies above the N = 0 axis,
  - Either solid B is partly soluble in the solvent or
  - An incompletely settled liquid has been withdrawn
- Solute A is infinitely soluble in solvent C, so that *x* and *y* may have values over the entire range from 0 to 1.0.
- The tie lines such as line FH are not vertical (for the reasons discussed before)
- The curve *DFE* represents the separated solid under conditions actually to be expected in practice.
  - **Example**: System soybean oil (A)-soybean meal (B)-hexane (C), where the oil and hexane are infinitely soluble.



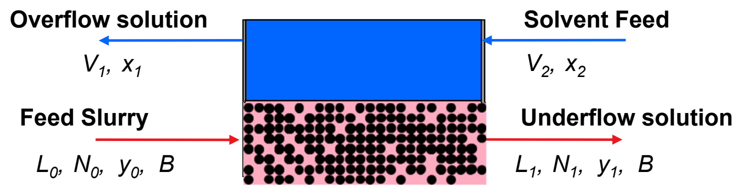
Equilibrium Phase Diagram for Leaching

- Solute A has a limited solubility x_A in solvent C.
- No clear solution stronger than x_A can be obtained, so that the tie lines joining slurry and saturated solution must converge.
- In this case any mixture M to the right of line PS will settle to give a clear saturated solution S and a slurry U whose composition depends on the position of M.
- Point T represents the composition of pure solid solute after drainage or settling of saturated solution.
- Since the tie lines to the left of PS are shown vertical, no adsorption occurs, and overflow liquids are clear



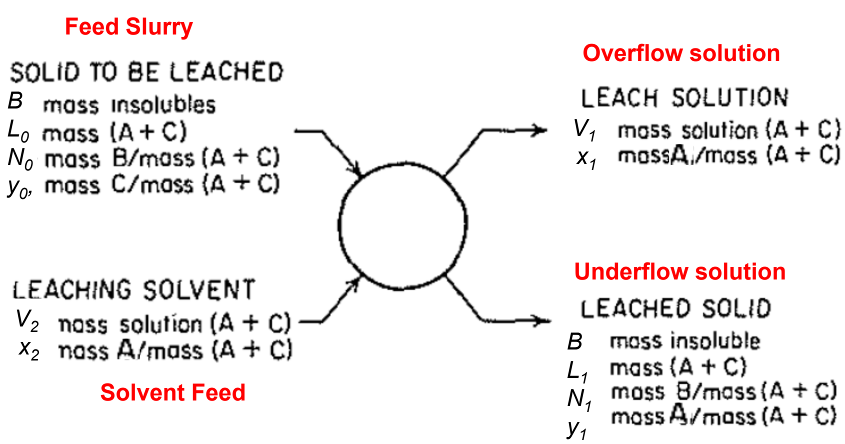
Single-Stage Leaching

Process flow



V	**Mass of overflow solution (A+C)**
L	**Mass of liquid (A+C) in slurry solution = (B/N)**
B	**Mass of dry, solute – free solid (insoluble solid) = N×L**
N	**Mass of dry-insoluble solid B/Mass of solution retained (L) = B/L = B/(A+C)**
x_A	**Composition of A at overflow solution = Mass A/(A+C)**
y_A	**Composition of A at slurry (underflow) solution = Mass A/(A+C)**

Single-Stage Leaching



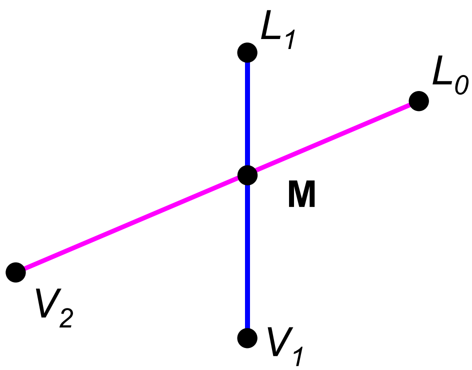
Single-Stage Leaching

- Material balance is divided into 3 parts:

$$L_0 + V_2 = L_1 + V_1 = M$$ Solute and solvent (Total solution) balance (A+C balance)

$$L_0 y_{A0} + V_2 x_{A2} = L_1 y_{A1} + V_1 x_{A1} = M x_{AM} \quad (\text{Comp. A balance})$$

$$B = N_0 L_0 + 0 = N_1 L_1 + 0 = N_M M$$ Dry insoluble solid (B) balance



→Lever-arm rule:

- L_0, V_2 and M must located on the same straight line and mixture point M is between L_0 and V_2.
- L_1,V_1 and M must located on the same straight line and mixture point M is between L_1 and V_1.

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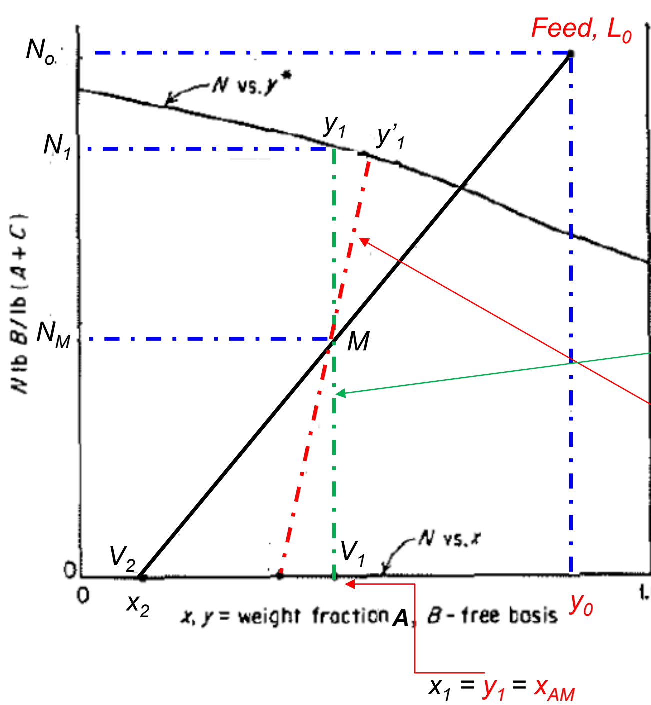
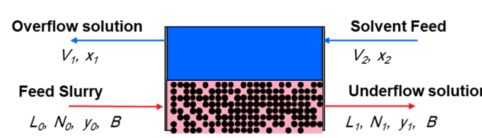
Single-Stage Leaching





Ideal stage

In a real stage there may be an additional inefficiency owing to short time of contact.

The stage efficiency= $(y_0 – y'_1) / (y_0 - y_1)$

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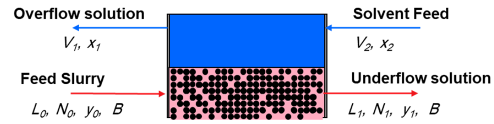
Example

In a single–stage leaching of soybean oil from flaked soybeans with hexane, 100 kg of soybean containing 20 wt% oil is leached with 100 kg of fresh hexane solvent. The mass ratio of insoluble solid to solution for the slurry underflow is essentially constant at 1.5 kg insoluble solid/kg solution. Calculate the amounts and compositions of the overflow and the underflow slurry leaving the stage.



- Feed slurry = 100 kg containing 20 wt% oil
- L_0 = (0.2)100= 20 kg A solute (oil)
- B = (1.0-0.2) 100= 80 kg insoluble solid
- N_0 = B/L_0 = 80/20=4.0 kg solid/kg solution (oil)
- Entering pure solvent, V_2 = 100 kg C (solvent), x_2=0
- N_1 = 1.5 kg B/kg solution
- L_1 =? ;V_1 =? ;x_1 =y_1?

Solution

Solution using mass balances:

- Total solution balance: $L_0 + V_2 = L_1 + V_1 = M \rightarrow M = 120\,\text{kg}$
- Solute A MB balance:

$$L_0 y_0 + V_2 x_2 = M x_M$$

$$(20)(1.0) + (100)(0) = (120) x_M$$

$$x_M = 0.167 = x_1 = y_1$$

- Solid balance:

$$B = N_1 L_1 = N_M M \rightarrow 80 = (1.5)(L_1) \rightarrow L_1 = 53.3\,\text{kg}$$

$$V_1 = M - L_1 = 120 - 53.3 = 66.7\,\text{kg}$$

Solution Cont.d

Solution using phase diagram and lever rule:

- Find coordinate at L_0.
  - Mass of A = 0.20 x 100 A = 20 kg
  - Mass of B = 0.80 x 100 B = 80 kg
  - Mass of C = 0 kg C = 0 kg

$$y_{A0} = \frac{A}{L_0} = \frac{A}{A+C} = \frac{20}{20+0} = 1.0$$

$$N_0 = \frac{B}{L_0} = \frac{B}{A+C} = \frac{80}{20+0} = 4.0$$

  - Coordinate for L_0 $(y_{A0}, N_0) = (1.0, 4.0)$

Solution Cont.d

- Find coordinate at V_2.
  - Mass of A = 0 A = 0 kg
  - Mass of B = 0 B = 0 kg
  - Mass of C = 100 kg C = 100 kg

$$x_2 = \frac{A}{V_2} = \frac{A}{A+C} = \frac{0}{0+100} = 0$$

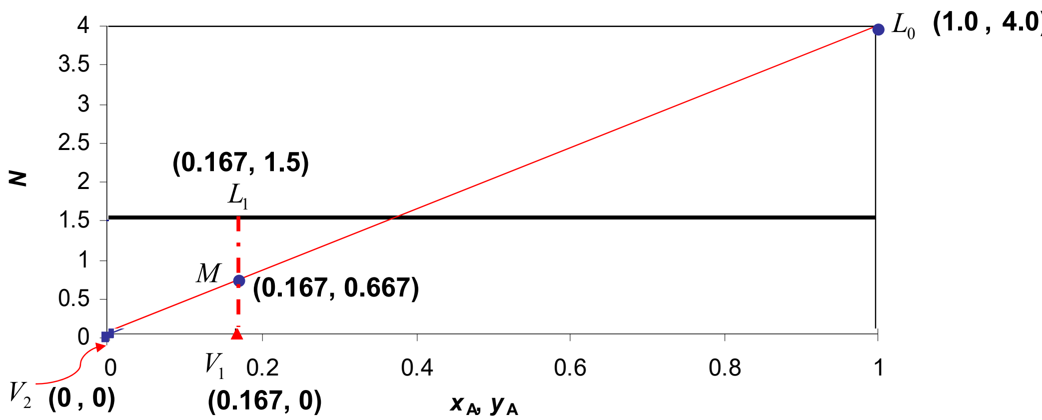
$$N_2 = \frac{B}{V_2} = \frac{B}{A+C} = \frac{0}{0+100} = 0$$

- Coordinate for V_2 $(x_2, N_2) = (0, 0)$
- Draw the N-xy phase diagram
- Locate L_0 and V_2 points on the phase diagram

Solution Cont.d

- Solute A MB balance:

$$L_0 y_0 + V_2 x_2 = M x_M$$
$$(20)(1.0) + (100)(0) = (120) x_M$$
$$x_M = 0.167 = x_1 = y_1$$



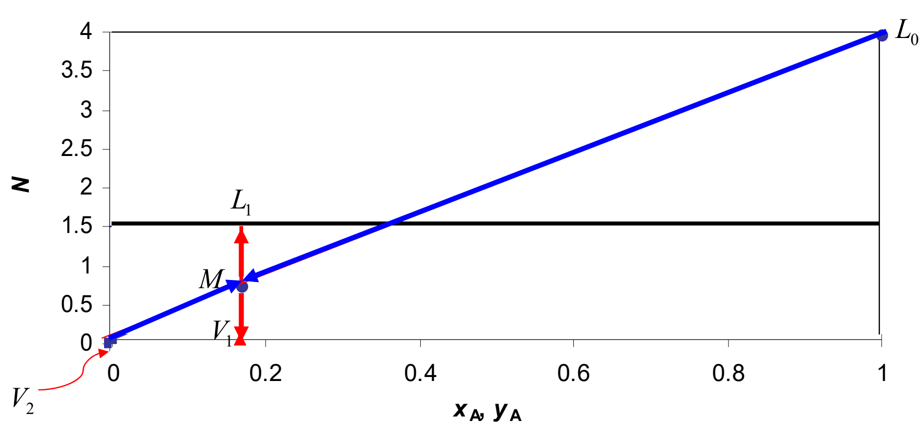
Solution Cont.d

or

- Solid balance: $B = N_o L_o = N_m M \Rightarrow 80 = N_m(120)$

$$N_m = 0.667$$

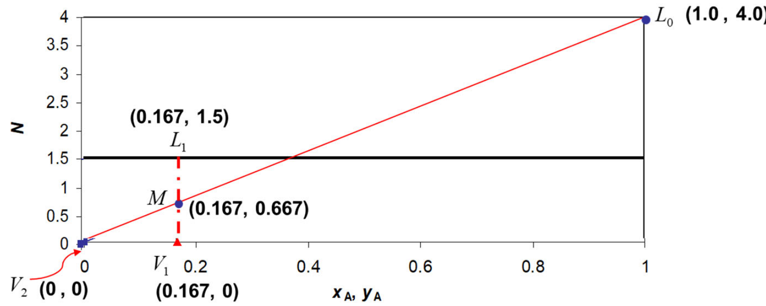
Note that $x_m = 0.167$



Solution Cont.d

$$\frac{Segment\ L_1V_1}{Segment\ L_1M} = \frac{\boldsymbol{M}}{V_1} \rightarrow$$

$$V_1 = \boldsymbol{M} \times \frac{Segment\ L_1M}{Segment\ L_1V_1}$$



And

$$\frac{Segment\ L_1V_1}{Segment\ MV_1} = \frac{\boldsymbol{M}}{L_1} \rightarrow L_1 = \boldsymbol{M} \times \frac{Segment\ MV_1}{Segment\ L_1V_1}$$

Then a ruler can be used to measure the line segments and calculate the quantities of $\boldsymbol{L_1}$ and $\boldsymbol{V_1}$.

Solution Cont.d

Since this is a vertical line, we can use the y-dimension units as measurements, or grid measurements units instead of ruler!

$$Segment\ L_1V_1 = 1.5 \ \ and\ \ Segment\ L_1M = 1.5 - 0.667 = 0.833$$

$$V_1 = 120 \times \frac{0.833}{1.5} = 66.64\ kg$$

And

$$Segment\ L_1V_1 = 1.5\ and\ \ Segment\ \ MV_1 = 0.667$$

$$V_1 = 120 \times \frac{0.667}{1.5} = 53.36\ kg$$

Summary

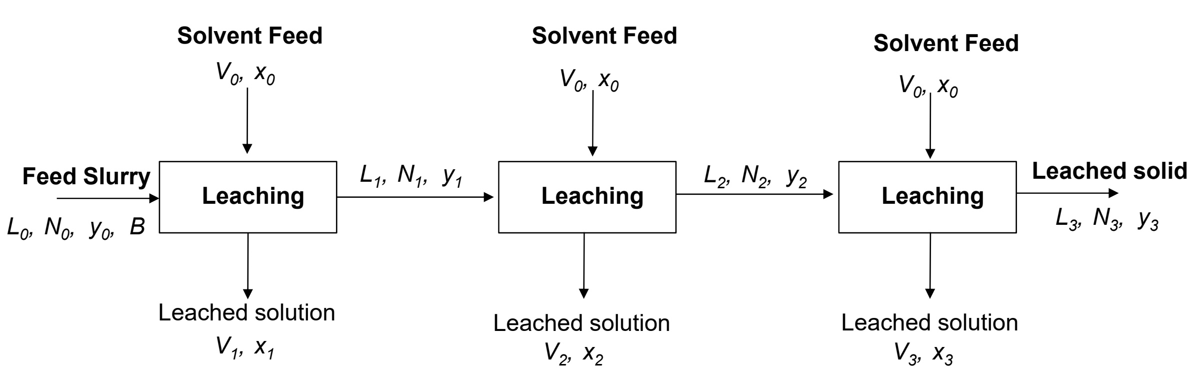
The solution of this problem can be presented graphical equilibrium system as follows:

1. The equilibrium curve is plotted as the *N versus y* for the underflow.
2. Remember $x_M = x_1 = y_1 =$ **0.167**. Plot a vertical tie line connecting these points from the *N* versus *y* curve to the *N* = 0 line (x-axis). Note that concentration of L_1, i.e., y_1 is on the *N* versus *y* curve and we find $N_1 =$ **1.5**. Concentration of V_1, i.e., x_1 is on the *N* = 0 line. Concentration of *M*, i.e., x_M is somewhere between the two at $N_M =$ **0.667**
3. Plot L_0 point at calculated coordinates $(y_0, N_0) =$ **(1.0 , 4.0)**
4. Plot V_2 point at calculated coordinates $(x_2, N_2) =$ **(0 , 0)**
5. Draw a line joining L_0, *M* and V_2. These should lie on a straight line, so where this line intersect the vertical tie line should be point *M* with coordinates $(x_M, N_M) =$ **(0.167, 0.667)**
6. Draw a line joining L_1, *M* and V_1. Remember from Step 2 above, L_1 coordinates are (y_1, *N* versus *y* intersection) = **(0.167, 1.5)** and V_1 coordinates are (x_1, *N*=0) = **(0.167, 0)** .

Multistage Cross-current Leaching

- Additional solute can be extracted by treating the leached solids from stage 1 with fresh batch of solvent as shown in the Figure for a 3-stage cross-current leaching unit.

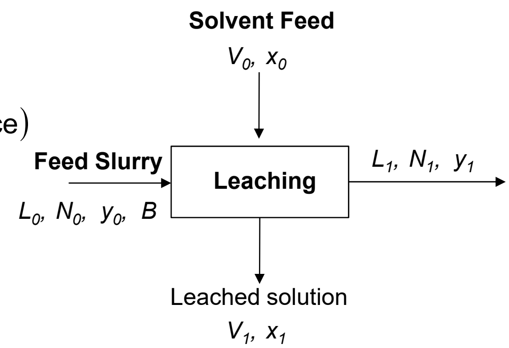
Process flow



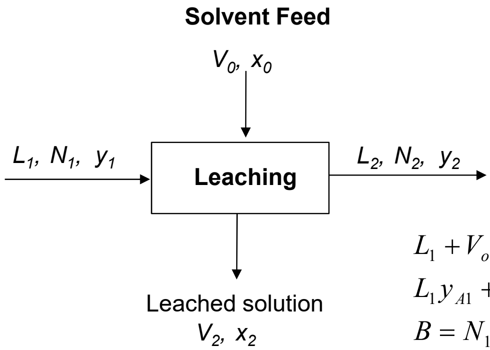
Multistage Cross-current Leaching

Stage 1

$$L_0 + V_o = L_1 + V_1 = M_1 \quad \text{(Total solution balance)}$$
$$L_0 y_{A0} + V_o x_{Ao} = L_1 y_{A1} + V_1 x_{A1} = M_1 x_{AM1} \quad \text{(Comp. A balance)}$$
$$B = N_0 L_0 + 0 = N_1 L_1 + 0 = N_{M1} M_1 \quad \text{Dry solid (B) balance}$$



Stage 2

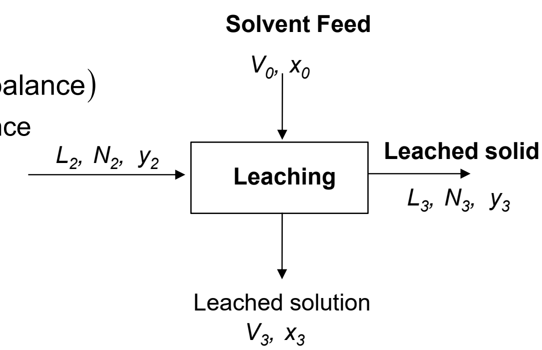


$$L_1 + V_o = L_2 + V_2 = M_2 \quad \text{(Total solution balance)}$$
$$L_1 y_{A1} + V_o x_{Ao} = L_2 y_{A2} + V_2 x_{A2} = M_2 x_{AM2} \quad \text{(Comp. A balance)}$$
$$B = N_1 L_1 + 0 = N_2 L_2 + 0 = N_{M2} M_2 \quad \text{Dry solid (B) balance}$$

Multistage Cross-current Leaching

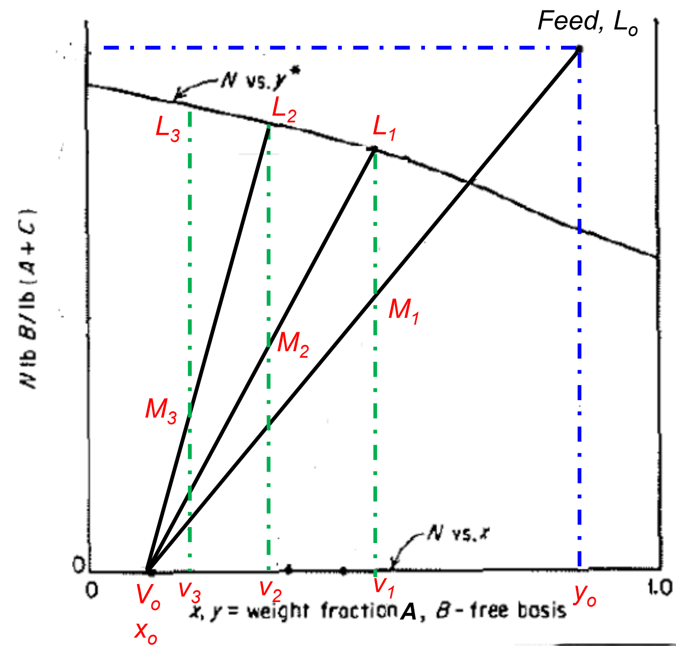
Stage 1

$$L_2 + V_o = L_3 + V_3 = M_3 \quad \text{(Total solution balance)}$$
$$L_2 y_{A2} + V_o x_{Ao} = L_3 y_{A3} + V_3 x_{A3} = M_3 x_{AM3} \quad \text{(Comp. A balance)}$$
$$B = N_2 L_2 + 0 = N_3 L_3 + 0 = N_{M3} M_3 \quad \text{Dry solid (B) balance}$$



Multistage Cross-current Leaching

- The calculations for subsequent stages are similar to those for the first stage with the leached solids from any stage becoming the feed for the next stage.



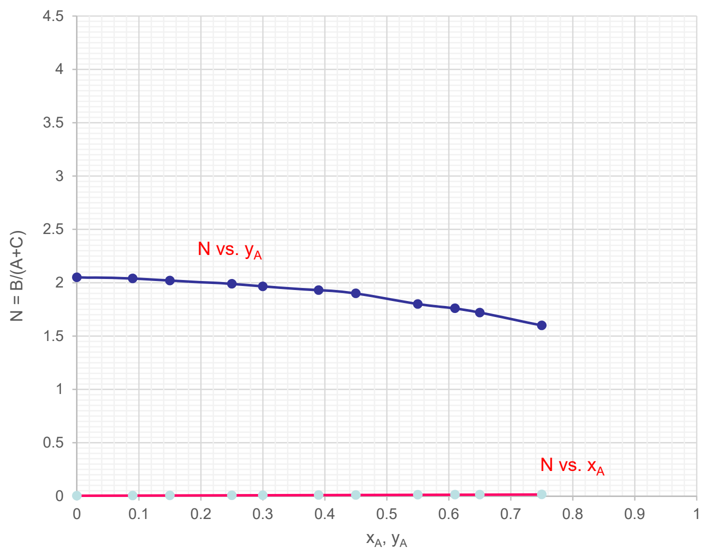
Task

One thousand kilograms of crushed oil seeds (19.5% oil, 80.5% meal) is extracted with 1500 kg of 'pure' hexane in a batch extraction vessel. Calculate the fraction of the oil extracted. The equilibrium data are given as

x_A	0	0.09	0.15	0.25	0.3	0.39	0.45	0.55	0.61	0.65	0.75
N	0.003	0.0045	0.0054	0.007	0.0078	0.0092	0.01	0.012	0.013	0.013	0.015
y_A	0	0.09	0.15	0.25	0.3	0.39	0.45	0.55	0.61	0.65	0.75
N	2.05	2.04	2.02	1.99	1.965	1.93	1.9	1.8	1.76	1.72	1.6

Answer: Fraction of the oil extracted= 76.6%

Task



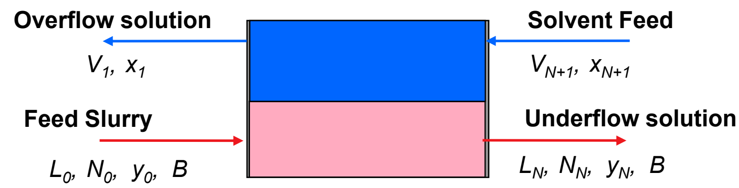
Task

- In the previous task, if instead of using the solvent at one time, it is planned to carry out the leaching in three stage cross-current unit using one third of the solvent (i.e. 500 kg hexane) in each stage. Calculate the fraction of oil that can be extracted.

Answer: Fraction of the oil extracted= 89 %

Multi – Stage Counter Current Leaching

Process flow



V	**Mass of overflow solution**	x_A	**Composition of A at overflow solution**
L	**Mass of liquid in slurry solution**	y_A	**Composition of A at slurry solution**
B	**Mass of dry, solute – free solid.**		

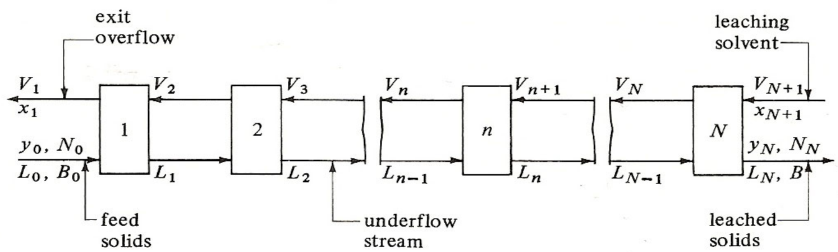
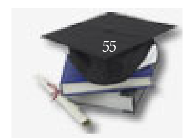


FIGURE 12.10-1. *Process flow for countercurrent multistage leaching.*



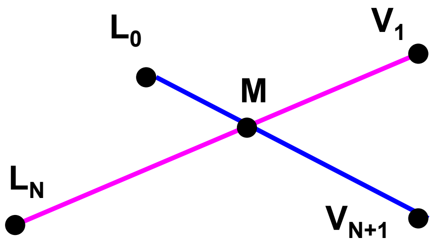
Multi – Stage Counter Current Leaching

- The ideal stages are numbered in the direction of the solids or underflow stream.
- The solvent (C) – solute (A) phase or *V* phase is the liquid phase that overflows continuously from stage to stage counter-currently to the solid phase, and it dissolves solute as it moves along.
- The slurry phase L composed of inert solid (B) and liquid phase of A and C is the continuous underflow from each stage.
- Composition of V – denoted by x
- Composition of L – denoted by y
- Assumption:
  - The solid B is insoluble and is not lost in the liquid V phase.
  - The flow rate of solid is constant throughout the process

Multi – Stage Counter Current Leaching

Apply solution mass balance over the overall system:

$$L_0 + V_{N+1} = V_1 + L_N = M$$



→Lever-arm rule:

- L_0, V_{N+1} and M must located on the same straight line and mixture point M is between L_0 and V_{N+1} .
- L_N,V_1 and M must located on the same straight line and mixture point M is between L_N and V_1 .

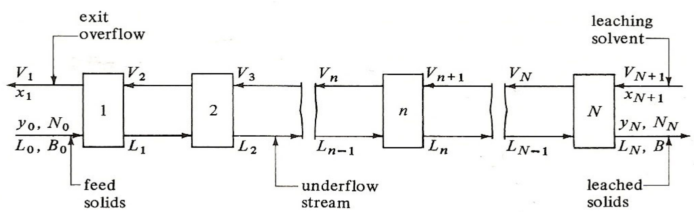


FIGURE 12.10-1. *Process flow for countercurrent multistage leaching.*

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Multi – Stage Counter Current Leaching

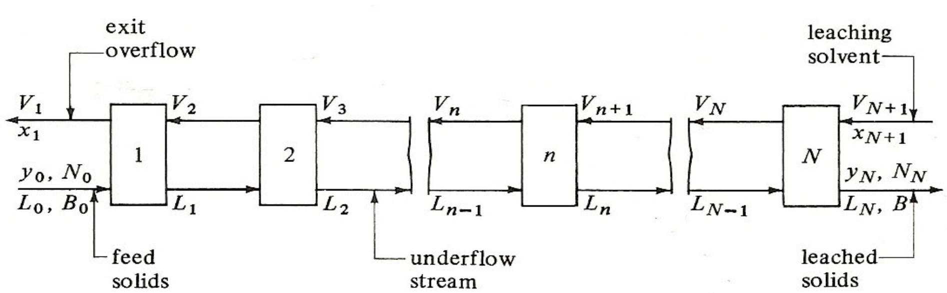

FIGURE 12.10-1. *Process flow for countercurrent multistage leaching.*

$L_0 + V_{N+1} = L_N + V_1 = M$ Solute and solvent (Total solution) balance (A+C balance)

$L_0 y_{A0} + V_{N+1} x_{AN+1} = L_N y_{AN} + V_1 x_{A1} = M x_{AM}$ (Comp. A balance)

$B = N_0 L_0 = N_N L_N = N_M M$ Dry insoluble solid (B) balance

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Multi – Stage Counter Current Leaching

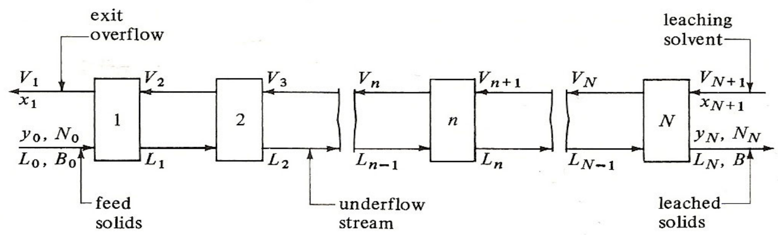


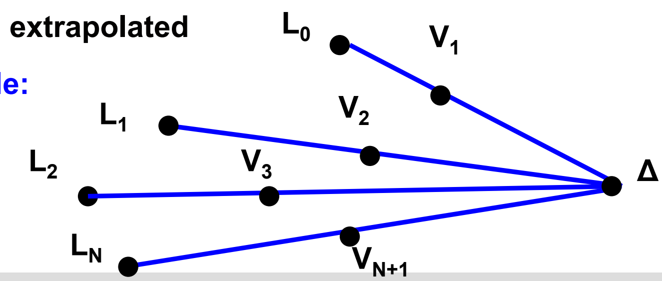
FIGURE 12.10-1. *Process flow for countercurrent multistage leaching.*

Apply solution mass balance on each stage gives:

$$V_1 - L_0 = V_2 - L_1 = V_3 - L_2 = ... = V_N - L_{N-1} = V_{N+1} - L_N = Constant = \Delta$$

Δ: operating point is extrapolated

Using Lever-arm rule:



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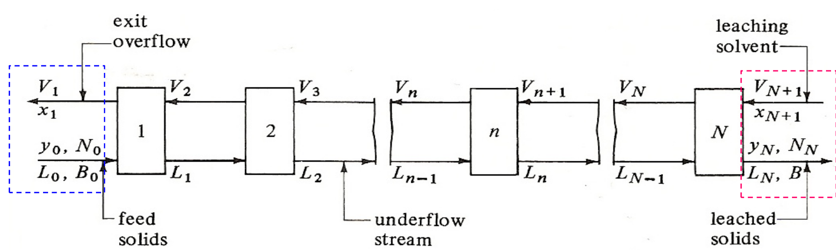
Multi – Stage Counter Current Leaching



For a balance on solute *A*

$$x_{A\Delta}\Delta = x_{A1}V_1 - y_{Ao}L_o = x_{An+1}V_{n+1} - y_{An}L_n = x_{AN+1}V_{N+1} - y_{AN}L_N$$

$$x_{A\Delta} = \frac{y_{Ao}L_o - x_{A1}V_1}{L_o - V_1} = \frac{y_{AN}L_N - x_{AN+1}V_{N+1}}{L_N - V_{N+1}}$$

where $x_{A\Delta}$ is the x coordinate of the operating point Δ.

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Multi – Stage Counter Current Leaching

A balance given on solids gives

$$B = N_o L_o = N_N L_N = N_M M = N_\Delta \Delta$$

$$N_\Delta = \frac{B}{L_o - V_1} = \frac{N_o L_o}{L_o - V_1}$$

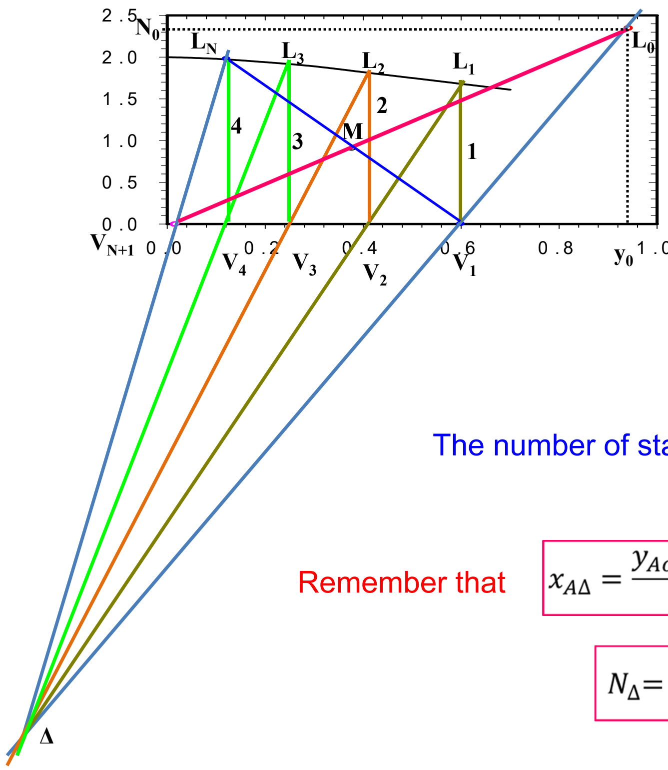
where N_Δ is the x coordinate of the operating point Δ.

Multi – Stage Counter Current Leaching

General procedure:

1. Connect V_{N+1} and L_0 points with a line.
2. Locate the mixture point M using overall total mass balance and overall component balance on A.
3. V_1 is located on the N versus x equlibrium curve (x-axis)
4. L_N is located on the N versus y equlibrium curve.
5. Draw a straight line through M and through V_1 or L_N point. The intersection with the equilibrium curve give the V_1 or L_N point.
6. Locate the operating point Δ:
   - Connect V_{N+1} through L_N and extrapolate
   - Connect V_1 through L_0 and extrapolate
   - Cross lines at operating point Δ

6. In general: connect V_n and L_n via equilibrium tie lines.



The number of stages: N=4

Remember that $x_{A\Delta} = \dfrac{y_{Ao}L_o - x_{A1}V_1}{L_o - V_1}$

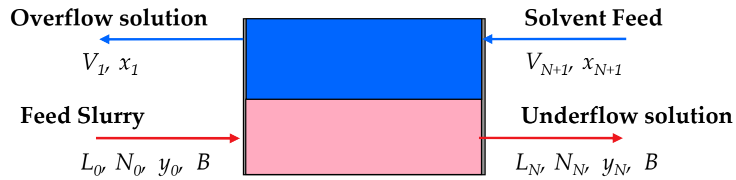
$$N_\Delta = \frac{B}{L_o - V_1}$$

Example

A continuous countercurrent multistage system is to be used to leach oil from vegetable seed meal by benzene solvent (C). The process is to treat 2000 kg/h of inert solid meal (B) containing 800 kg/hr oil/ (A) and also 50 kg/hr benzene (C). The inlet flow per hour of fresh solvent mixture contains 1310 kg benzene and 20 kg oil. The leached solids are to contain 120 kg oil/hr. Equilibrium data are tabulated below as *N* kg inert solid B/kg solution and y_A kg oil A/kg solution. Calculate the amounts and concentrations of the stream leaving the process and the theoretical number of stages.

N	y_A	N	y_A
2.00	0	1.82	0.4
1.98	0.1	1.75	0.5
1.94	0.2	1.68	0.6
1.89	0.3	1.61	0.7

Solution



- Information given:
  - Feed slurry (L_0):

    A = 800 kg/h   B = 2000 kg/h   C = 50 kg/h

  - Entering solvent (V_{N+1})

    A = 20 kg/h   B = 0 kg/h   C = 1310 kg/h

  - Underflow solution (L_N):

    A =120 kg/h   B = 2000 kg/h   C = ?? kg/h

Solution Cont.d

➢ Find coordinate at L_0.

- Mass of A = 800 kg/h
- Mass of B = 2000 kg/h
- Mass of C = 50 kg/h
- L_0= A+C = 800+50=**850 kg solution/h**

$$y_{A0} = \frac{A}{L_0} = \frac{A}{A+C} = \frac{800}{800+50} = \frac{800}{850} = 0.94$$

$$N_0 = \frac{B}{L_0} = \frac{B}{A+C} = \frac{2000}{800+50} = \frac{2000}{850} = 2.35$$

- Coordinate for L_0 (y_{A0}, N_0) = (0.94 , 2.35)

Solution Cont.d

- Find coordinate of Solvent Feed at V_{N+1}.
  - Mass of *A* = 20 kg/h
  - Mass of *B* = 0 kg/h
  - Mass of *C* = 1310 kg/h
  - V_{N+1}= A+C = 1310+20=**1330 kg solution /h**

$$x_{N+1} = \frac{A}{V_{N+1}} = \frac{A}{A+C} = \frac{20}{20+1310} = \frac{20}{1330} = 0.015$$

$$N_{N+1} = \frac{B}{V_{N+1}} = \frac{B}{A+C} = \frac{0}{20+1310} = 0$$

  - Coordinate for V_{N+1} (x_{N+1}, N_{N+1}) = (0.015 , 0)

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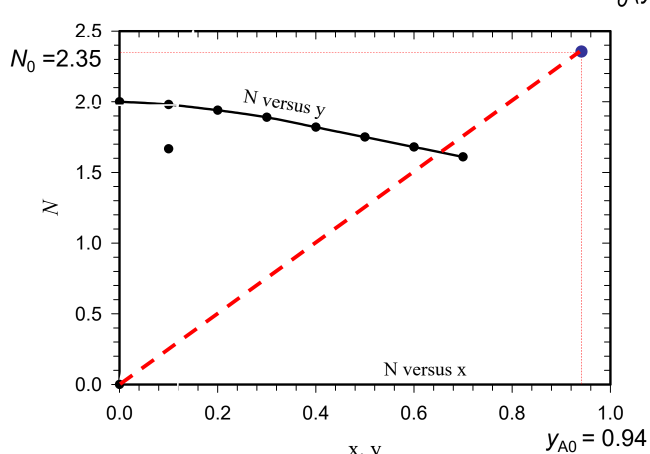
Solution Cont.d

L_0 (y_{A0}, N_0) = (0.94 , 2.35)



$V_{N+1}$$(x_{N+1}, N_{N+1})$ = (0.015 , 0)

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Solution Cont.d

- From material balance calculations:

  - Total solution balance:

$$L_0 + V_{N+1} = L_N + V_1 = M$$
$$L_0 + V_{N+1} = M$$
$$850 + 1330 = M \qquad M = 2180\,\text{kg}$$

  - Component A balance:

$$L_0 y_{A0} + V_{N+1} x_{N+1} = M x_{AM}$$
$$(850)(0.94) + (1330)(0.015) = (2180) x_{AM}$$
$$x_{AM} = 0.376$$

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Solution Cont.d

- From material balance calculations:

  - Solid balance:

$$B = N_0 L_0 = N_N L_N = N_M M$$
$$N_0 L_0 = N_M M$$
$$(2.35)(850) = N_M (2180) \qquad N_M = 0.916$$

  - Coordinate for *M* $(x_M, N_M) = (0.376, 0.916)$

- Plot coordinate M in the graph.

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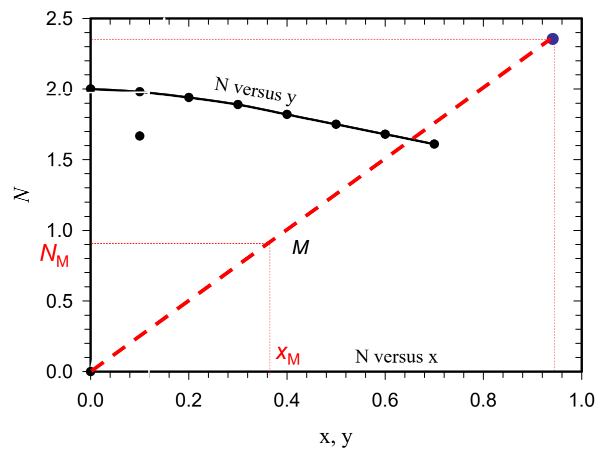
Solution Cont.d



Solution Cont.d

- Find coordinate at L_N
  - Mass of A = 120 kg/h
  - Mass of B = 2000 kg/h
  - Mass of C = ?? kg/h

Mass of solution (A+C) = 120 + ????

L_N = (A+C) = ????

We know that the point L_N lies on the N versus Y_A

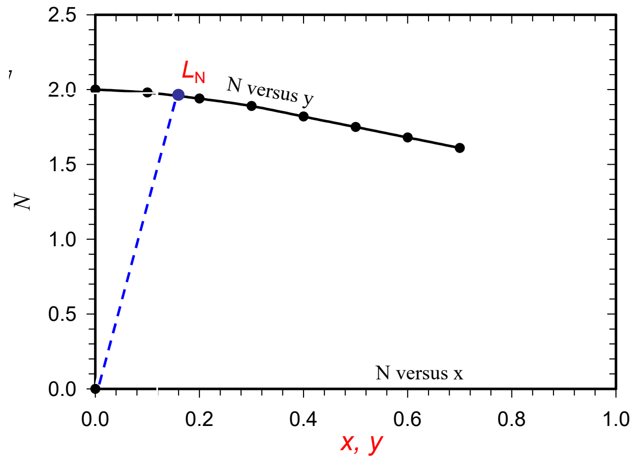
Solution Cont.d

Place an arbitrary point L_N to aid the explanation

The ratio of N/y for this point is

$$\text{Slope of dotted line, } \frac{N_N - 0}{y_N - 0} = \frac{N_N}{y_N}$$

$$N_N = \frac{B}{L_N} \qquad y_N = \frac{A}{L_N}$$

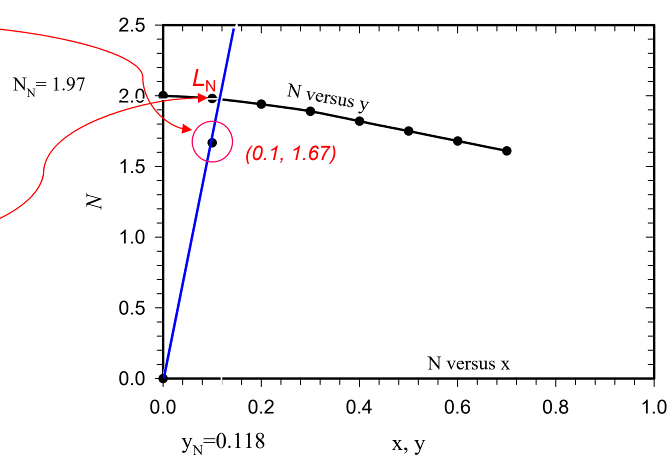


$$\frac{N_N}{y_N} = \frac{\frac{B}{L_N}}{\frac{A}{L_N}} == \frac{\frac{B}{(A+C)_N}}{\frac{A}{(A+C)_N}} = \frac{B}{A} = \frac{2000}{120} = 16.67 \qquad \rightarrow \qquad N = 16.67y$$

Solution Cont.d

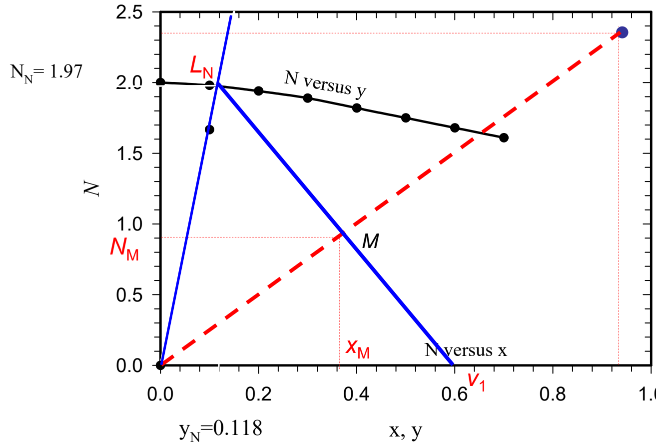
- If we draw a line from (0,0) with slope 16.67, it will the N vs y curve at the actual point L_N.
- If y = 0.1, N = 16.67 x 0.1 = 1.67

➢ Thus, the location of L_N is at which, y_N=0.118 and N_N=1.97 kg B/kg solution



Solution Cont.d

- Construct line from point L_N to point M until it cross at x – axis. Point at x – axis = V_1



- Coordinate for V_1

  $(x_1 , N_1) = (0.592 , 0)$

- Coordinate for L_N

  $(y_N , N_N) = (0.12 , 2.0)$

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Solution Cont.d

$$y_N = \frac{A}{L_N} \qquad L_N = \frac{A}{y_N} = \frac{120}{0.118} = 1016kg$$

$L_N = (A + C)$ therefore $C = (L_N - A) = 1016 - 120 = 896$ kg

Next we can do total mass balance around stage 1,

$$L_N + V_1 = M \quad ⇨ \quad V_1 = M - L_N = 2180kg - 1016kg = 1164kg$$

And then component V balance,

$$L_N y_N + V_1 x_1 = M x_M \quad ⇨ x_1 = \frac{Mx_M - L_N y_N}{V_1} = \frac{2180 \times 0.38 - 896 \times 0.118}{1164} = 0.62$$

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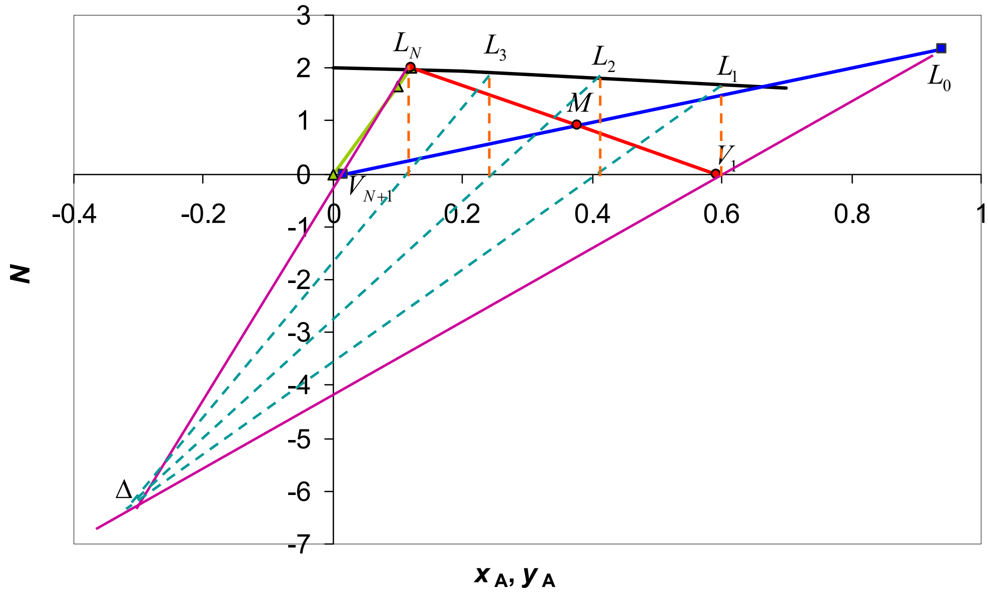
Solution Cont.d



Task

Countercurrent Multistage Leaching of Halibut Livers. Fresh halibut livers containing 25.7 wt % oil are to be extracted with pure ethyl ether to remove 95% of the oil in a countercurrent multistage leaching process. The feed rate is 1000 kg of fresh livers per hour. The final exit overflow solution is to contain 70 wt % oil. The retention of solution by the inert solids (oil-free liver) of the liver varies as follows (C1), where N is kg inert solid/kg solution retained and y_A is kg oil/kg solution:

N	y_A	N	y_A	N	y_A
4.88	0	2.47	0.4	1.39	0.81
3.50	0.2	1.67	0.6		

Calculate the amounts and compositions of the exit streams and the total number of theoretical stages.

Answer: L_N=175.8, y_{AN}=0.073, V_1=348.8 kg/hr, N_0=2.89, 6.1 stages needed

Solution

- Feed slurry (L_0): A = 0.257 (1000) = 257 kg/h B = 1000-257= 743 kg/h C = 0 kg/h

  y_{oA} = 1.0 N_o = B/A = 2.98

- Entering solvent (V_{N+1}): A = 0 kg/h B = 0 kg/h C = ??? kg/h

- overflow (V_1): A = 0.95 (257) = 244.94 kg/h B = 0 kg/h C = ?? kg/h

  x_{1A} = 0.7 N = 0.0

A = 0.95 (257) = 244.94 kg/h = 0.7 V_1 ➔ **V_1 = 349.9 kg/h**

- Underflow solution (L_N): A =257- 244.94 = 12.85 kg/h B = 743 kg/h C = ?? kg/h

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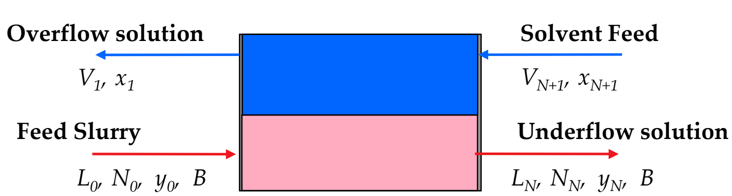
Solution Cont.d

$$x_{A\Delta} = \frac{y_{Ao}L_o - x_{A1}V_1}{L_o - V_1} = \frac{y_{AN}L_N - x_{AN+1}V_{N+1}}{L_N - V_{N+1}}$$

$$\rightarrow x_{A\Delta} = \frac{y_{AN}L_N - x_{AN+1}V_{N+1}}{L_o - V_1} = \frac{0.05(257) - 0}{257 - 348.8} = -0.14$$

$$N_\Delta = \frac{B}{L_o - V_1} = \frac{N_o L_o}{L_o - V_1}$$

$$\rightarrow N_\Delta = \frac{743}{257 - 348.8} = -8.1$$



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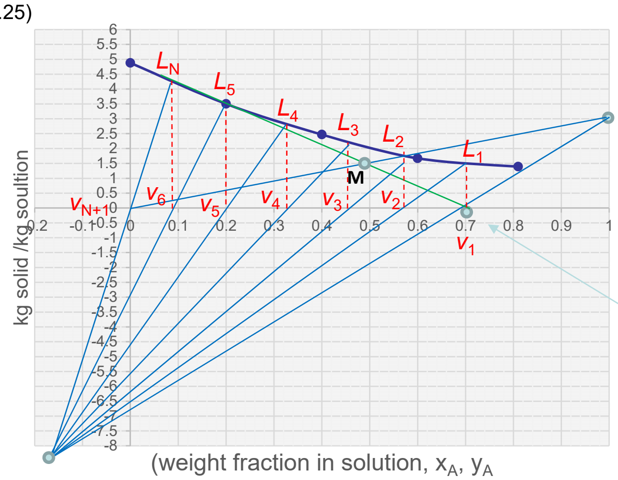
Solution Cont.d

- Coordinate for L_N

$(y_{AN}, N_N) = (0.073, 4.25)$

- Feed slurry (L_0):

$y_{oA} = 1.0$ $\quad N_o = 2.98$



- overflow (V1):

$x_{1A} = 0.7$ $\quad N = 0.0$

(-0.14, -8.1)
$x_{A\Delta}$, N_Δ)

$B = N_0 L_0 = N_N L_N$ → 743 =4.4 L_N → L_N =174.8 kg/h

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Solution Cont.d

$$x_{A\Delta} = \frac{y_{AN} L_N - x_{AN+1} V_{N+1}}{L_o - V_1} = -0.14$$

, y_{AN} = 0.073 and L_N **=174.8 kg/h**

→ V_{N+1} = 265.9 kg/h

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Types of Equipments for Leaching

- **Fixed – Bed Leaching**
- **Moving Bed Leaching**
- **Agitated Solid Leaching**

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Fixed – Bed Leaching

- Used in beet sugar industry and is also used for extraction of tanning extracts from the tanbark, extraction of pharmaceuticals from barks and seeds and other processes.
- Figure 12.8-1 shows a typical sugar beet diffuser or extractor.

- The cover is removable so that sugar beet slices called ***cossettes*** can be dumped into the bed.
- Heated water at 344 K to 350 K flows into the bed to leach out the sugar.
- The leached sugar solution flows out the bottom onto the next tank in series.
- About 95% of the sugar in beets is leached to yield an outlet solution from the system of about 12 wt%.

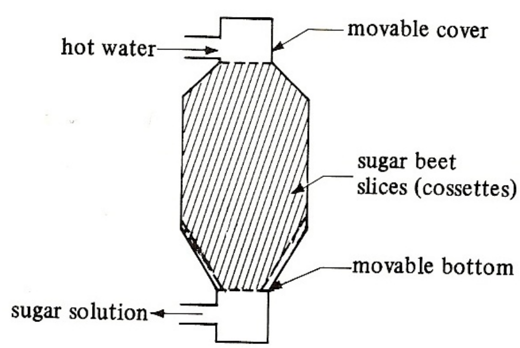

FIGURE 12.8-1. *Typical fixed-bed apparatus for sugar beet leaching.*

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Moving – Bed Leaching

- There are number of devices for stagewise countercurrent leaching where the bed or stages moves.
- Used widely in extracting oil from vegetable seeds such as cottonseeds, peanuts and soybeans.
- The seeds are usually dehulled first, sometimes precooked, often partially dried and rolled or flaked.
- The solvents used are particularly hydrocarbons such as hexane and the final solvent – vegetable solution called miscella may contain some finely divided solids.

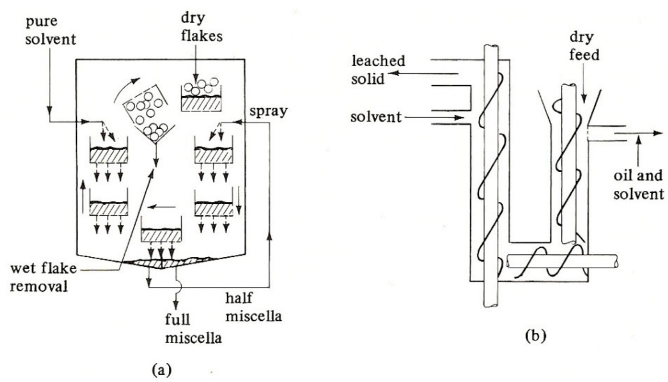

FIGURE 12.8-2. *Equipment for moving-bed leaching: (a) Bollman bucket-type extractor, (b) Hildebrandt screw-conveyor extractor.*

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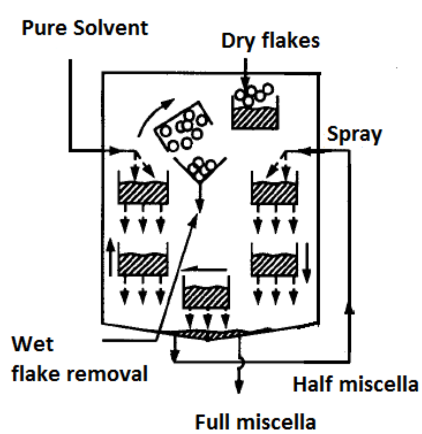
Moving – Bed Leaching

Bollman extractor:

- It contains a bucket elevator in a closed casing.
- Dry solids are added at the upper right side to a Perforated basket or bucket such as soybeans.
- The solids are sprayed with appropriate amount of half miscella as they travel downward.
- Half miscella is the intermediate solvent containing some extracted oil and some small solid particles.
- The liquid downward through the moving buckets and is collected at the bottom as the strong solution or full miscella.



Bollman bucket-type extractor

- The buckets moving upward on the left are leached counter currently by fresh solvent sprayed on the top bucket.
- The wet flasks are dumped as shown in the Figure and removed continuously.

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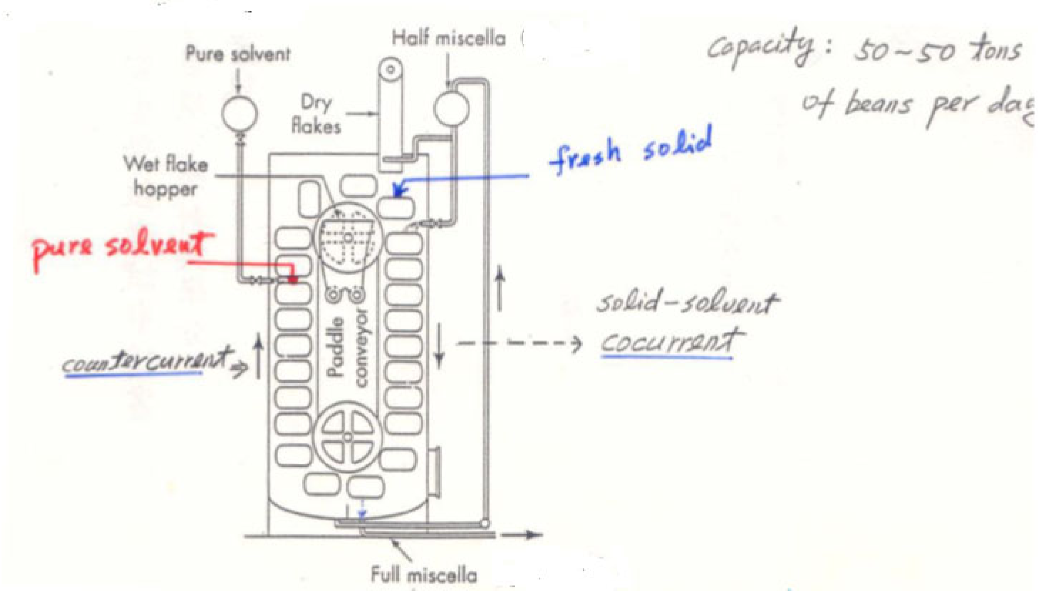
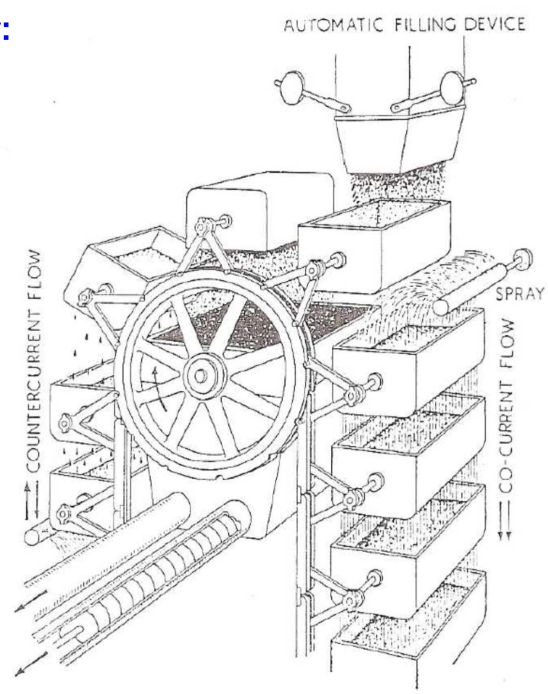
Moving – Bed Leaching

Bollman extractor:

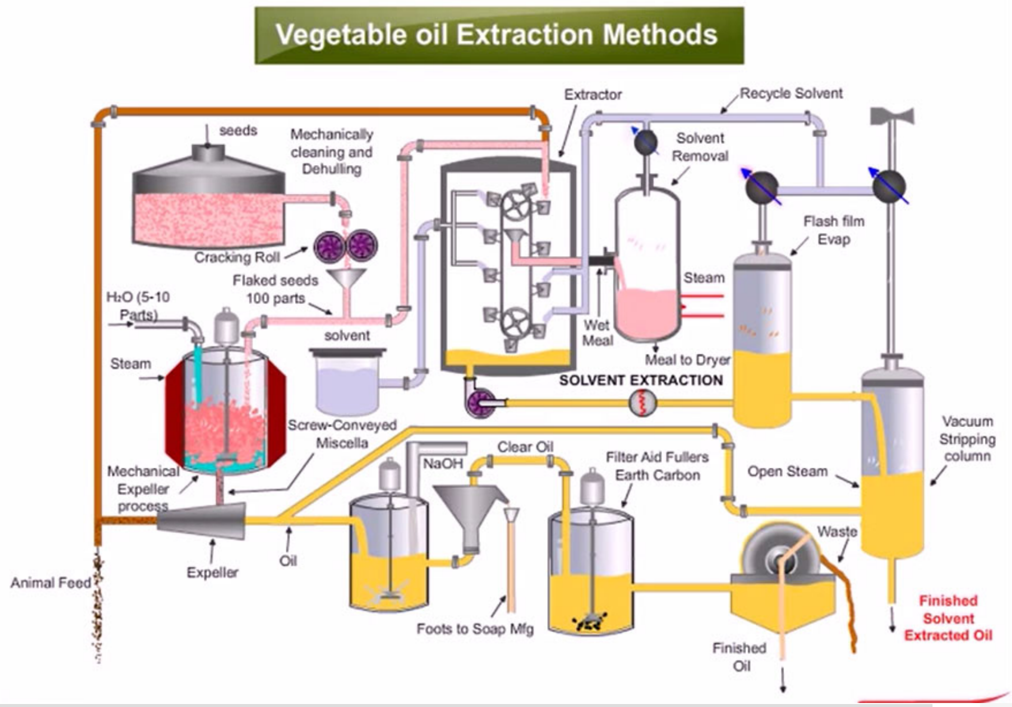


Moving – Bed Leaching

Bollman extractor:



Moving – Bed Leaching



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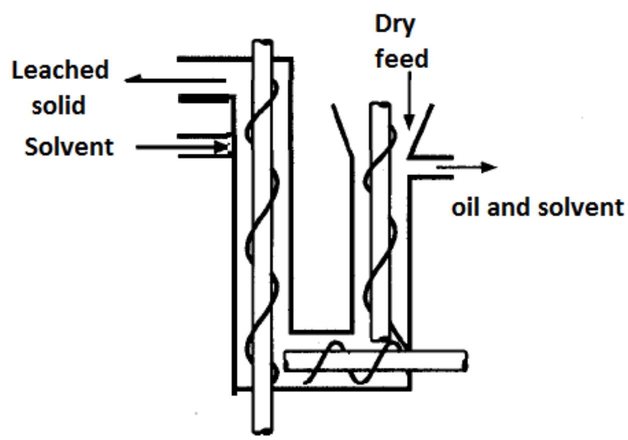
Moving – Bed Leaching

Hildebrandt extractor:

- Consists of three screw conveyers arranged in a U shape.
- The solids are charged at the top right conveyed downward, across the bottom , and then up the other Leg .
- The solvent flows counter currently to the solid.



Hildebrandt screw-conveyor extractor

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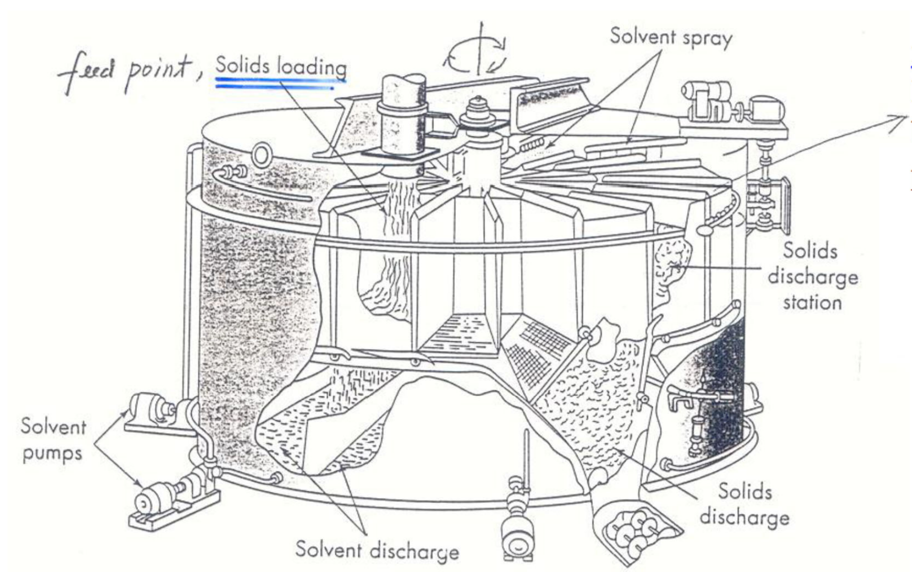
Moving – Bed Leaching

Rotocel Extractor

- A horizontal basket is divided into walled compartments with a floor that is permeable to the liquid.
- The basket rotates slowly about a vertical axis.
- Solid are admitted to each compartment at feed point.
- The compartments then pass a number of solvent sprays, a drainage section and a discharge point.
- To give countercurrent extraction, the fresh solvent is fed only to the last compartment before the discharge point.

Moving – Bed Leaching

Rotocel Extractor



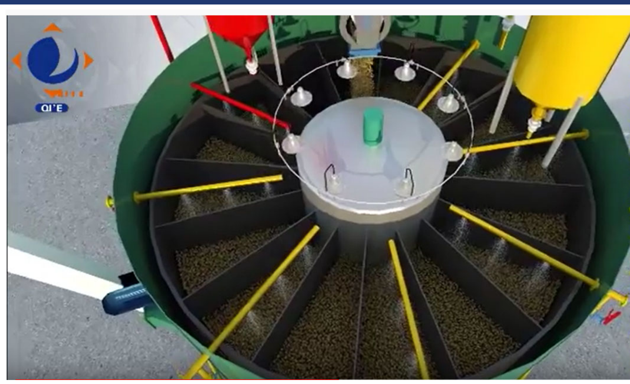
fresh solvent feed , last compartment before the discharge point

Moving – Bed Leaching

Rotocel Extractor

Moving – Bed Leaching

Rotocel Extractor



Moving – Bed Leaching

Rotocel Extractor

Agitated Solid Leaching

- When the solid can be ground fine abou 200 mesh (0.074 mm), it can kept in suspension by small amounts of agitation.
- Continuous countercurrent leaching can be accomplished by placing the number of agitator in series, with setttling tanks or thickeners between each agitator.
- Sometimes thickeners are used as combination contactor – agitators and settlers – shown in Figure 12.8-3.

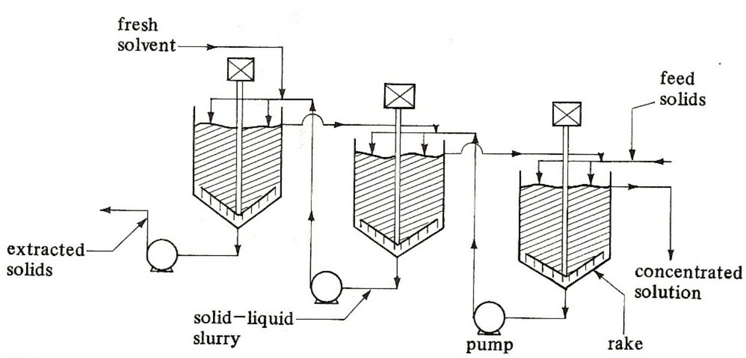


FIGURE 12.8-3. *Countercurrent leaching using thickeners.*

Kennedy Extractor

Kennedy Extractor