Leaching

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Leaching is the extraction of a solute from an insoluble solid by contact with a liquid solvent. The following are examples of leaching:

- Extraction of sugar from sugar cane by contact with hot water
- Extraction of mineral salts from crushed ores
- Extraction of oil from halibut livers by contact with benzene

- Extraction of sodium hydroxide from insoluble carbon carbonate by contact with water
- Extraction of coffee from coffee grounds by contact with hot water.

1 The Leaching Operation

Leaching is carried out by contacting the solid with the liquid solvent and separating the liquid extract from the solid. Various types of equipment can be used to carry out this operation, but the most common is to do the contacting in a *digester* followed by separating the extract in a *decanter*, as Figure 1 shows.



Figure 1. Typical leaching equipment.

The equipment shown in Figure 1 constitutes one stage of leaching. The complete operation usually requires a battery consisting of a number of stages arranged in countercurrent flow, as shown in Figure 2.



Figure 2. Cascade of countercurrent stages for leaching.

2 Balances on Leaching Battery

A leaching operation involves three different components, the *solid*, the *solute*, and the *solvent*. The solid is insoluble in the solvent so its flow does not change through the battery. It is the same from stage to stage and the same from inlet to outlet. The solid, however carries with it some of the solution (solute + solvent) from stage to stage. The flow of the solution carried by the solid is called the **underflow**. The extract that flows from stage to stage is called the **overflow** and consists of solution only, solute plus solvent. To avoid mistakes in the leaching material balances it is very important to understand the following:

The UNDERFLOW is the solution, solute plus solvent, carried by the solid from stage to stage. IT DOES NOT INCLUDE THE SOLID.

As in any other operation the design calculations start with the over-all material balances around the entire battery. Consider the schematic of the battery shown in Figure 3:



Figure 3. Over-all balances on the cascade

The notation of Figure 3 is as follows:

- L = flow of underflow, solvent plus solute
- V = flow of overflow
- x = composition of solute in the underflow
- y =composition of solute in the overflow

Subscript a is normally used on the end of the cascade where the solid enters and the extract exits, and subscript b for the end where the solid exits and the solvent enters. The compositions x and y are of the solute in the solutions, excluding the solid.

As the solid flow through the cascade is constant, there is no need to write a balance on the solid, so this leaves us two components, the solute and the solvent, and two balances. Usually we write a total balance and a solute balance, but remember that the total balance does not include the solid.

Total balance: $L_a + V_b = L_b + V_a$ (1)

Solute balance:	$L_a x_a + V_b y_b = L_b x_b + V_a y_a$	(2)
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2.1 Underflow Function.

One additional relationship in leaching is the dependence of the underflow L on its composition x. The underflow is the liquid solution entrained in the solids leaving the decanter (see Figure 1). In general the underflow is a function of its composition as the composition affects the density and viscosity of the solution, that is, the underflow may be larger the higher its composition. The underflow function, U(x)must be may be determined experimentally and а function of the properties of the solid, of the solution, and of the equipment used to separate the overflow (the decanter). As will be discussed shortly, under certain conditions and on the appropriate flow basis the underflow may be assumed to be constant, that is, to remain the same from stage to stage.

2.2 Specifications.

With two balance equations plus the underflow function and eight variables, solution of the over-all balances requires five specifications. The feed flow and composition, L_a and x_a , and the solvent composition, y_b , are standard specifications in a design problem. Another specification is either the solute recovery or the exit underflow composition, x_b ; finally we have either the solvent inlet rate V_b or the exit extract composition, y_a , but one of these is usually a design variable left to the discretion of the designer.

After the compositions and flows of the outlet streams are determined from the over-all balances, the number of required stages can be determined from stage to stage calculations using the operating and equilibrium lines. These will be discussed next.

3 Operating Line

As usual, the operating line is obtained from balances around n stages in the cascade, as follows:





Once again we can write a total balance and a solute balance:

Total balance:
$$L_a + V_{n+1} = V_{a+1} L_n$$
 (3)

Solute balance:
$$L_a x_a + V_{n+1} y_{n+1} = V_a y_a + L_n x_n$$
 (4)

Combine the two equations and solve for y_{n+1} , to obtain the equation of the operating line:

$$y_{n+1} = \frac{L_n}{V_{n+1}} x_n + \frac{V_a y_a - L_a x_a}{V_{n+1}}$$
(5)

The operating line goes through the points (x_a, y_a) and (x_b, y_b) . When the underflow *L* can be assumed constant, by Equation (3), the overflow *V* is also constant and the operating line is straight. When the underflow varies from stage to stage the operating line is curved and Equation (5) must be used to plot intermediate points on the line.

4 Equilibrium Line

In leaching the equilibrium assumption is that the solution entrained in the solid leaving a stage has the same composition as that of the extract leaving the stage, that is,

$$y_n = x_n \tag{6}$$

Thus the equilibrium line is always a straight line with a slope of 1.0. Notice that this is valid independent of the basis used on the composition (mole fraction, weight fraction, molar ratio, weight ratio, or concentration).

5 Flow Basis

As with other stagewise operations, the determination of the number of required stages is simplified when both the equilibrium and operating lines are straight, because the number of stages can then be determined with the Kremser equation. Given that, in leaching, the equilibrium line is straight for any flow basis, it behooves a smart chemical engineer to select a flow basis in which the operating line is straight, if possible. For example, if the volume of solution entrained in the solid is approximately constant, then a volumetric flow basis (liters/min) results in a constant underflow and thus a straight operating line.

It is important to realize that the flow basis determines the basis for the composition. If the basis for the composition does not match the basis for the flow, the calculation of the terms Fx in the balance equations will not result in the rate of solute as it must. For example, F in liters/min must be multiplied by x in kg/liter (concentration) to obtain kg/min of solute.

The following are some of the common flow and composition bases that can be selected to carry out the material balances. It is important to realize that the flow basis and the composition basis must match:

Flow basis	Composition basis	
Weight flow	Weight fraction	
Molar flow	Mole fraction	
Solute-free weight flow	Weight ratio	
Solute-free molar flow	Molar ratio	
Volume flow	Concentration	

On the solute-free basis the flow is in rate of solvent per unit time and the composition is in units of solute per unit of solvent, for example, kg of solvent/hr and kg solute/kg solvent. The conversions of total flow to solute-free basis and weight fraction to weight ratio are as follows:

$$L' = L(1 - x)$$
 $X = \frac{x}{1-x}$

$$V' = V(1 - y)$$
 $Y = \frac{y}{1-y}$

Where L' and V' are the flows in weight of solvent per unit time, and X and Y are the weight ratios in weight of solute/weight of solvent.

6 Balances on the First Stage

The feed to the battery does not follow the underflow relationship because it is not the exit stream from a decanter as those in the battery. Therefore the feed point is not on the operating line determined for the rest of the stages making it necessary to carry out separate balance calculations for the first stage, as follows:



Figure 5. Balances on the First Stage.

The first stage is assumed to be an equilibrium stage, as are the others:

$$\mathbf{x}_1 = \mathbf{y}_a$$

The underflow from stage 1 follows the underflow relationship: $L_1(x_1)$. Then

$$V_2 = V_a + L_1 - L_a$$
 $y_2 = \frac{V_a y_a + L_1 x_1 - L_a x_a}{V_2}$

The determination of stages 2 to N can now be carried out by stage-to-stage calculations, McCabe-Thiele graphical procedure or, if the equilibrium and operating lines are straight, the Kremser equation.

Example 1. Design of a Leaching Battery

The feed to a leaching battery consists of 10,000 kg/hr containing 70 weight% insoluble solid and the balance solute. Pure solvent at the rate of 9,000 kg/hr is used to extract 95% of the solute. Each kg of solid carries (1.1 + 0.25x) kg of solution of solute mass fraction x. Determine the number of equilibrium stages required to carry out the desired extraction.

Solution: The first thing to do is to draw a schematic of the battery showing all the problem data.



Figure 6. Schematic and problem data for Example 1.

The feed consists of the following:

Solids: 0.70(10,000 kg/hr) = 7,000 kg/hr Solute: 0.30(10,000 kg/hr) = 3,000 kg/hr Solvent: 10,000 - 7,000 - 3,000 = 0 kg/hr

Total solution in feed: $L_a = 3.000 + 0 = 3,000 \text{ kg/hr}$ Composition of the solution in the feed: $x_a = 3,000/3,000 = 1.0$

So the composition of the solute in the solution in the feed is 100 weight% (not 30 weight%). This is because the solids are excluded and there is no solvent in the feed. (You must study this until you are sure you understand it.)

The underflow function given in the statement of the problem is in kg of solid and is normally obtained experimentally from measurements on the equipment. So the flow of underflow solution leaving each stage is calculated by:

 $L_n = (7,000 \text{ kg/hr}) (1.1 + 0.25x_n)$

From the recovery we can determine the flow of unrecovered solute leaving with the solids:

$$L_b x_b = (1 - 0.95) L_a x_a = 0.05 (3,000) (1.0) = 150 \text{ kg/hr}$$

Because the underflow from the last stage follows the underflow function, $L_b = 7,000(1.1 + 0.25x_b)$, and we can write:

 $L_b x_b = 7,000 (1.1 + 0.25 x_b) x_b = 150 \text{ kg/hr}$

We can solve this equation to obtain: $x_b = 0.0194$ Then, $L_b = 7,000(1.1 + 0.25 \times 0.0194) = 7,730$ kg/hr

From the total balance: $V_a = L_a + V_b - L_b$ = 3,000 + 9,000 - 7,730 = 4,270 kg/hr And from the solute balance:

$$y_a = \frac{L_a x_a + V_b y_b - l_b x_b}{V_a} = \frac{(3,000)(1.0) + (9,000)(0) - (7,730)(0.0194)}{4,270} = 0.667$$

We must now carry out the balances around the first stage:



Figure 7. Balances on stage 1 for Example 1.

Assuming equilibrium stages, $x_1 = y_a = 0.667$.

From underflow function:
$$L_1 = 7,000(1.1 + 0.25x_1)$$

= 7,000(1.1 + 0.25×0.667)
= 8,870 kg/hr

Total balance on stage 1:
$$V_2 = V_a + L_1 - L_a$$

= 4,270 + 8,870 -3,000
= 10,140 kg/hr

Solute balance on stage 1:

$$y_2 = \frac{V_a y_a + L_1 x_1 - L_a x_a}{V_2} = \frac{4,270(0.667) + 8,870(0.667) - 3,000(1.0)}{10,140} = 0.568$$

Now we are ready to determine stages 2 to N. First let us check if the operating line can be assumed to be approximately straight. We do this by comparing the slope of the operating line at both ends:

$$L_1/V_2 = 8,870/10,140 = 0.875$$
 $L_b/V_b = 7,730/9,000 = 0.858$

As the slope changes by less than 2% from one end to the other, we can assume the operating line is approximately straight. As the equilibrium line is also straight, we can use the Kremser equation to estimate the required number of equilibrium stages after the first one. For leaching:

$$x_1^* = y_2 = 0.568$$
 $x_b^* = y_b = 0$

$$N = \frac{ln\left(\frac{x_1 - x_1^*}{x_b - x_b^*}\right)}{ln\left(\frac{x_1 - x_b}{x_1^* - x_b^*}\right)} + 1 = \frac{ln\left(\frac{0.667 - 0.568}{0.0194 - 0}\right)}{ln\left(\frac{0.667 - 0.0194}{0.568 - 0}\right)} + 1 = 13.4$$

Notice that the Kremser equation is applied for stages 2 to N, that is, it does not include the first stage. This is the reason the "1" is added.

Had we determined the required number of equilibrium stages by the McCabe-Thiele method, we would have obtained the following plot:



Figure 8. McCabe-Thiele Diagram for Example 1.

As expected, the number of equilibrium stages is a bit over 13, showing that the Kremser equation is a good approximation in this example. The graph shows why the first stage must be considered separately.

Example 2. Design of a Sugar Cane Mill

The mills in a sugar Factory are fed 30,000 kg/hr of cane containing 10 weight% fiber, 13 weight% sugar, and the balance water. Pure hot water is used to extract the sugar. The fiber, called *bagasse*, is insoluble in the water. It leaves each stage carrying 1.2 kg of sugar solution per kg of fiber on a sugarfree basis. Design the battery to recover 99.5% of the sugar in the cane and to produce a final raw juice containing 12 weight% sugar.



Figure 9. Rendition of a sugar mill with three mills.

Solution: The first step, as always, is to draw a schematic of the mill showing the problem data.



Figure 10. Schematic of Sugar Mill of Example 2.

The feed consists of:

Fiber (insoluble solid): 0.10(30,000 kg/hr) = 3,000 kg/hr Sugar (solute): 0.13(30,000 kg/hr) = 3,900 kg/hr Water (solvent): 30,000 - 3,000 - 3,900 = 23,100 kg/hr

By the statement of the problem, the underflow is constant on a sugar-free (solute-free) basis, so the problem is easier to solve on this basis. The basis consists of measuring all flows on sugar-free basis, which is the water weight flow, and the compositions in weight ratios, or kg of sugar per kg of water. As follows:

Feed: $L_a' = 23,100 \text{ kg/hr}$ $X_a = 3,900/23,100 = 0.169$ Extract: $Y_a = 0.12/(1 - 0.12) = 0.136 \text{ kg sugar/kg water}$ Underflow: L' = 3,000U(X) = 3,000(1.2) = 3,600 kg/hr (constant)

From the recovery, the unrecovered sugar leaving in the bagasse is:

 $L_b'X_b = (1 - 0.995)L_a'X_a = 0.005(23,100)(0.169) = 19.5 \text{ kg/hr}$

Now, $L_b' = L' = 3,600 \text{ kg/hr}$, and $X_b = 19.5/3,600 = 0.00542$

From the solute balance: $V_a' = \frac{L_a' X_a + V_b' Y_b - L_b' X_b}{Y_a} = \frac{23,100(0.169) + 0 - 19.5}{0.136} = 28,600 \frac{\text{kg}}{\text{hr}}$

From the total balance: $V_b' = V_a' + L_b' - L_a'$ = 28,600 + 3,600 - 23,100 = 9,100 kg/hr

Because the underflow is constant on a sugar-free basis, the overflow is also constant on a sugar-free basis, that is,

$$V' = V_b' = 9,100 \text{ kg/hr}$$

Now that we have all the outlet flows and compositions and the solvent rate, we are ready to determine the required number of equilibrium stages. We start by doing the balances on stage 1:



Figure 12. Balances on Stage 1 for Example 2.

As stage 1 is an equilibrium stage, as are the others,

$$X_1 = Y_a = 0.136$$

Solute balance on stage 1:

$$Y_2 = \frac{V_a'Y_a + L'X_1 - L_a'X_a}{V'} = \frac{28,600(0.136) + 3,600(0.136) - 23,100(0.169)}{9,100} = 0.0522$$

As the underflow and overflow rates from stages 2 to N are constant on a sugar-free basis, the operating line is straight on this basis. In leaching the equilibrium line is straight on any basis (Y = X), so we can use the Kremser equation to determine the number of required equilibrium stages from stages 2 to N:

$$X_1^* = Y_2 = 0.0522$$
 $X_b^* = Y_b = 0$

$$N = \frac{\ln\left(\frac{X_1 - X_1^*}{X_b - X_b^*}\right)}{\ln\left(\frac{X_1 - X_b}{X_1^* - X_b^*}\right)} + 1 = \frac{\ln\left(\frac{0.136 - 0.0522}{0.00542 - 0}\right)}{\ln\left(\frac{0.136 - 0.00542}{0.0522 - 0}\right)} + 1 = 4.0$$

Thus four equilibrium stages are required.

The weight fractions of the outlet streams can be calculated from the weight ratios:

 $x_b = X_b/(1 + X_b) = 0.00542/1.00542 = 0.00539$

 $y_a = Y_a / (1 + Y_a) = 0.136 / 1.136 = 0.120$

The outlet flows in total solution weight rates are:

 $L_b = L_b'(1 + X_b) = 3,600(1.00542) = 3,620 \text{ kg/hr}$

$$V_a = V_a'(1 + Y_a) = 28,600(1.136) = 32,500 \text{ kg/hr}$$

It is very important to understand the following points:

- The balance calculations for this example can be carried out on a total solution flow basis and the corresponding compositions in weight fractions. The results will be exactly the same as the ones obtained here.
- Given the conditions from the problem statement, the operating line is not straight on the total solution flow basis, so if the weight fractions are used in the Kremser equation the resulting number of equilibrium stages will only be approximate.

• Exact results can be obtained by converting the weight fractions to weight ratios and then using the weight ratios in the Kremser equation.

If you are not fully convinced you should try it and see that you get a different result when you use the weight fractions and the same result when you use the weight ratios. This will be a good exercise.

Summary

These notes have presented the design of leaching batteries. It has been shown that what is characteristic of leaching is that the equilibrium line is always straight with a slope of 1.0. As this is true on any composition basis it is possible under certain conditions to pick the basis that produces a straight operating line thus simplifying the calculation of the required number of equilibrium stages.

Review Questions

- 1. Define the leaching operation.
- 2. What is the underflow? Does the underflow include the insoluble solids?
- 3. What is the *underflow function*? How it is normally obtained in practice?
- 4. How many over-all mass balances are independent in leaching?
- 5. How many specifications are required to solve the over-all mass balance equations? Enumerate them.

- 6. What is the equilibrium relationship in leaching? On what composition basis is it valid?
- 7. How is the basis for the stream flows and composition selected?
- 8. Why must separate balances be made on the first stage of a leaching battery?

Problems

1. Extraction of Oil from Meal. Oil is to be extracted from meal, an insoluble solid, using benzene as the solvent. The unit is to treat 1,800 kg/hr of meal consisting of 70 weight% meal and 30 weight% oil. The solvent enters at 855 kg/hr and contains 1.5 weight% oil and the balance benzene. It is desired to recover 85% of the oil in the feed. Experiments carried out with the battery have resulted in the relationship below between the solution retained by the solid (underflow) and the oil concentration of the solution.

 $U(x) = 0.49842 + 0.05845x + 0.16786x^2$ where U(x) is the kg of underflow per kg of solid and x is the weight fraction oil in the underflow. Draw a schematic of the battery showing all the problem data and design the battery to determine the required number of equilibrium stages. Report also the flows and compositions of all the streams in and out of the battery. State all assumptions.

2. Extraction of Coffee in a Countercurrent Leaching Battery. A countercurrent leaching battery is to extract coffee from coffee grounds using hot water. The feed, at 1,100 kg/hr, contains 42 weight% coffee and the balance insoluble solid (coffee grounds). It is desired to recover 99.5% of the coffee in the feed. The grounds retain 0.45 kg of solution per kg of insoluble solid and the extract leaving the battery is to contain 55 weight% coffee. Draw a schematic of the battery showing all the problem data and design the battery to determine the number of required equilibrium stages and the flows and compositions of all the streams in and out of the battery.

3. Leaching of NaOH from Calcium Carbonate Sludge. (Based on Problem 23.2, McCabe, Smith & Harriott, 7th ed., page 792.) It is desired to extract the sodium hydroxide from the sludge produced by the following reaction:

Na₂CO₃ + CaO + H₂O --> CaCO₃ + 2NaOH

The insoluble CaCO₃ carries with it 1.5 kg of solution per kg of CaCO₃ in flowing from one stage to the next. It is desired to recover 99% of the NaOH. The reactor products enter the battery with no excess reactants but with 0.6 kg of water per kg of CaCO₃. The solvent is pure water. **Draw a schematic of the battery showing all the problem data and d**etermine the composition of the extract from the battery and the required number of equilibrium stages. Hint: Notice that the sludge contains 1 kmole of CaCO₃ (MW = 100) and 2 kmole of NaOH (MW = 40).

4. Dewaxing of Paper with Kerosene. A countercurrent leaching battery is to be designed to extract wax from insoluble paper pulp using kerosene as the solvent. The feed consists of 90 kg of wax paper per hour containing 25 weight% wax and the balance pulp. Solvent is fed containing 0.05 weight% wax and the balance kerosene. It is desired to leave no more than 0.2 kg of wax per 100 kg of pulp in the extracted solid. The underflow leaving each stage contains 2 kg of solution per kg of pulp on a solute-free basis. Draw a schematic of the battery showing all the problem data and design the battery to determine the number of required equilibrium stages and the flows and compositions of all the streams in and out of the battery.

- 5. Countercurrent Leaching Battery for Copper Ore. (Based on McCabe, Smith & Harriott, 7th edition, Problem 23.1, page 792.) The feed to a countercurrent leaching battery is 8,500 kg/hr of roasted copper ore consisting of 21.5 weight% CuSO4 and the balance inert rock. It is desired to extract 95% of the CuSO4 using pure water as a solvent. Experimental data reveals that the underflow from each stage contains a constant amount of 1.8 kg of solution per kg of inert rock on a solute-free basis. If the solution exiting the battery is to contain 15weight% CuSO4, determine the required number of equilibrium stages. Draw a schematic of the battery showing all the problem data and report the flows and compositions of all the streams in and out of the battery.
- 6. Parallel Leaching Battery for Copper Ore. (Based on McCabe, Smith & Harriott, 7th edition, Problem 23.1, page 792) Using the feed rate and composition of the countercurrent battery of Problem 5 consider a parallel leaching battery with the same number of stages and feeding the amount of solvent in each stage as is fed to the countercurrent battery. Determine the composition of the final solution

carried by the rock, the composition of the combined extract from all the stages after they are mixed, and the recovery of $CuSO_4$. Assume the underflow relationship is the same.