Universiti Malaysia PAHANG
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Process Chem and Pharmaceutical Engineering 1

Solid Liquid Separation

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Chapter Description

- Aims
  - Solve problems related to extraction process by applying the formula relevant to specific unit operations

- Expected Outcomes
  - Apply the principles of leaching, filtration and drying
  - Discuss the components involved in settling, sedimentation, centrifugation together with their applications

- References
  - Principles of Mass Transfer and Separation Processes, Binay K. Dutta, PHI Learning Private Limited, 2009

Solid-liquid Separation

- **Solid-liquid separation**: The process of separation of soluble constituents of a solid material using a suitable solvent.
- Soluble material → the target product
- Inert solid residue → byproduct or solid waste
- E.g. extraction of oils from oil seeds, the oil is the product and the solid residue is a byproduct

- Solid-liquid extraction is also called leaching, a substantial part of the solid does not dissolve and the soluble matters diffuse out through the solid during solid-liquid contact.

- Application: widely used in the food industries

The overall process of liquid-solid extraction involves four steps:

1
- Close contact between the solid feed with the solvent

2
- Separation of the solution (or extract) from the exhausted solid

3
- Separation of the solvent (and the entrained solid, if any from the extract followed by purification of the product)

4
- Recovery of the solvent from the moist solid (by squeezing/pressing and drying to get a dry cake)

Classification of Solid-Liquid Extraction System

- It may involve different physicochemical mechanisms depending upon the characteristics of the **solid** like porosity, particle size, compactness, reactivity, solute content and **solvent**.

- If the particle size of the solid is not small, there may be substantial diffusional resistance to transport of the solute within the solid. Whenever possible, the solid is broken down or disintegrated into particles or flakes to enhance the rate of extraction.

- Sometimes, an acidic, alkaline or a complexing solution may be used for solubilisation of the target substances.

Solid-liquid extraction

Diffusional extraction

- The extraction of sugar from sugar beet. Sugar beets are cut into small pieces which are treated with warm water (70-75°C).

- Diffusion within the solid occurs through denatured cell walls and through the interstitial liquid. The solvent (water) also diffuses in the opposite direction, thus diluting the solute within the cells.

- The rate and time of extraction depend on:
  (a) thickness of the pieces
  (b) the 'effective diffusivity' of the solute in the solid
  (c) the concentration of sugar in the extracting liquor

Washing extraction

- If the solid particle size is pretty small, the solid-phase diffusional resistance becomes negligibly small. In such a case, extraction virtually becomes a process of washing the solid with the solvent.

- An example is extraction of oil from ‘flakes’ of oil seeds.

- Flakes are pretty thin. During the flaking process, much of the cells are ruptured and the diffusional resistance for transport of the solute within the solid becomes small.

- In washing extraction, a substantial amount of solvent is retained in the slurry after washing

Leaching

- This involves dissolution of one/ more substances from solid particles accompanied by chemical reaction(s).
- An acid, alkali or the solution of a complexing chemical is commonly used for solubilizing the target materials
- Leaching of ores (oxides, carbonates, sulphides) is a major step in hydrometallurgy
    - Overflow – concentration of the solute in the clear liquid
    - Underflow – fraction of the liquid in the slurry

- Chemical extraction

Functionally similar to leaching but usually refers to recovering solutes from solids of organic nature.

Example, recovery of gelatin from animal bones using an alkaline solution is an example of chemical extraction

Factors Affect the Rate of Solid-Liquid Extraction

- Physical Characteristics of the Solid

The rate of extraction becomes faster if the particle size of the solid is small or if the solid is porous. Since rigid solids like metal ores and most compact solids dissolve slowly, such materials are crushed and ground

If the solute is distributed within an insoluble matrix of the ‘inert’ solid, it gradually dissolves and diffuses out leaving a porous structure. In some cases, the solid may be available as slurry/ paste. If the solute has an affinity for the insoluble solid, it may remain partly adsorbed to it

The type of the solid matrix: porous or non-porous

- Solvent

A solvent in which the solute has a high solubility is preferred. But it should not dissolve the undesired solids

The solvent should have a low viscosity, a low boiling point and should be non-toxic and reasonably cheap

It is easier to remove a low-boiling solvent from the product liquor by ‘flash vaporization’

- Temperature

Diffusivity of the solute in the solid as well as its miscibility with the solvent increases with temperature. Also, the viscosity of the solvent becomes less at an elevated temperature and it can penetrate into the interstices of the solid more easily. So the extraction rate increases with temperature

Heat sensitivity of the solute, volatility of the solvent and possible thermal effects on the inert or residual solid (e.g., solubility, degradation, change in consistency, etc.) limit the temperature of extraction

- Agitation

Intense agitation increases the solid-liquid mass transfer coefficient and increases the rate of extraction but may disintegrate the solid as well, causing a settling problem

However, if the solid phase offers the controlling resistance, the effect of agitation on the rate of extraction is not pronounced

Fluid Flow Through Porous Media

- The liquid flows through the open space between the particles such as pores or voids within the bed.
- Liquid flows over the surface of the solid packing cause the loss of friction and lead to a pressure drop.
- ↑ amount of solids inside the bed, ↑ larger surface area, ↑ friction, ↑ pressure drop.
- The volume fraction of the bed available for fluid flow → porosity or voidage.

Solid volume fraction concentration, C:

C = 1 – ε

In many solid-liquid separations porosity is often replaced by solid concentration (C)

$$porosity = \epsilon = \frac{volume\ of\ voids}{total\ bed\ volume}$$

Source: Filtration Fundamentals, Rushton, A., Ward, A. S., Holdich, R.G., 2008

Darcy's law

- **Darcy law**: the pressure loss is α to the flow rate of the fluid.
- Darcy's law : fluid flow through porous media.
- The constant of proportionality is dependent on the permeability, *k* (m^2) of the porous network.
- The resistance of fluid flow increases with the decrease of the permeability.

$$\frac{\Delta P}{L} = \frac{\mu q}{kA}$$

Where

ΔP = pressure drop

L = bed depth

μ = liquid viscosity

Q = volume flow rate

A = cross sectional-area of the bed

Permeability

- Factors that affect the permeability:
  a) particle size that making up the porous medium
  b) porosity
- Filter cake permeability is derived from :
  (i) Partcle shape
  (ii) Particle packing
  (iii) Particle size
  (iv) Porosity
  (v) Distribution rate at which the cake was formed
  (v) Concentration of the slurry being filtered
- The permeability equation (Kozeny,1927):

KOZENY CARMAN EQUATION

$$\frac{\Delta P}{L} = \mu \left(\frac{5(1-\varepsilon)^2 S_v^2}{\varepsilon^3} \right) \frac{q}{A}$$

Cake Filtration

- Filtration resulting in a filter cake takes place by a bridging mechanism over the surface pores within a filter medium, cloth, septum or support. This helps to prevent the medium from clogging with fine particles.

- **Filter medium**: initiating the filtration, have long-lasting effects on the filter cake structure and properties throughout the filtration cycle.

- The cake depth increases during filtration, due to deposition of solids at the filter cake surface.

- The change in cake depth is accompanied by changes in fluid flow rate and pressure differential, as filtration time increases.

$$\frac{\Delta P}{L} = \frac{\mu}{kA}\frac{dV}{dt}$$

Incompressible filtration

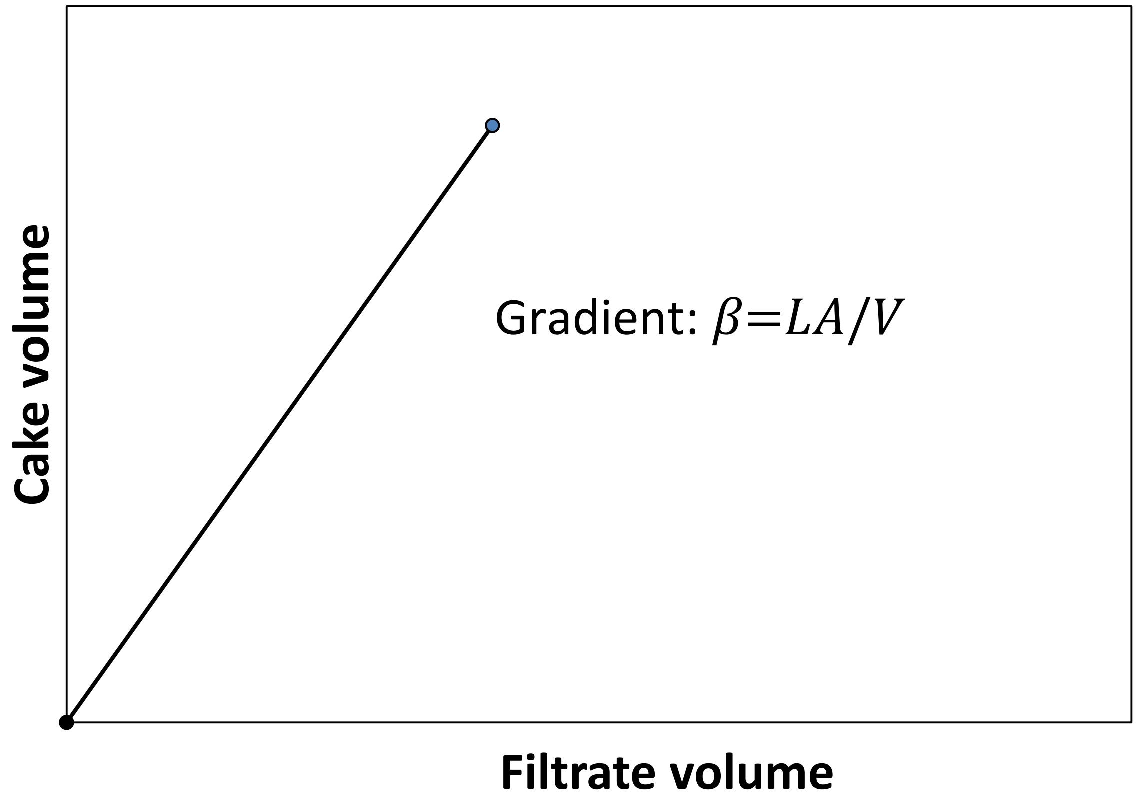
- The filtrate volume is uniform and constant with respect to each volume of suspension filtered.

- Filtering at constant pressure: the rate of cake deposition with respect to time will not be uniform because each new element of filter cake increases the total resistance to the passage of filtrate from the new cake layer. Thus the rate of filtration declines.

- The constant of proportionality β can be used to give an equation for cake depth, at any instant in time:

$$L = \frac{\beta V}{A} \quad \Rightarrow \quad \frac{dV}{dt} = \frac{A \Delta P k}{\beta V \mu}$$

Relationship between Cake and Filtrate Volume



Filter Media

- **Role of filter media**: to get a clean separation of particulate solids from a flowing fluid

- **Classification of filter media**:

  (a) design to recover a valuable solid product

  (b) use in the clarification of a fluid

Conclusion of The Chapter

Overall, this chapter discussed about the process of solid-liquid extraction, classification of solid-liquid extraction, factors affect the rate of solid-liquid extraction and filter media.

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Reference

[1] Principles of Mass Transfer and Separation Processes, Binay K. Dutta, PHI Learning Private Limited, 2009

[2] Filtration Fundamentals, Rushton, A., Ward, A. S., Holdich, R.G., 2008.