

Chapter I:

Separation of a heterogeneous mixture



Introduction:

Separation methods are essential in analytical chemistry, the chemical industry, and environmental sciences to isolate and purify different components of a mixture. These methods allow the separation of homogeneous or heterogeneous substances based on their chemical nature or physical properties. They are widely used to purify chemicals, separate impurities, or extract valuable components.

❖ Reminders and definitions

■ Fluids:

Definition: A fluid is any substance that can flow, meaning it has the ability to take the shape of its container. Fluids include both liquids and gases: **Examples:** Water (liquid), air (gas).

■ Solution:

Definition: A solution is a homogeneous mixture composed of two or more substances where the solute is completely dissolved in the solvent. The particles in a solution are very small and not visible to the naked eye: **Examples:** Salt dissolved in water, sugar in tea.

■ Electrolyte Solution:

Definition: An electrolyte solution is a solution in which the solute dissociates into ions when dissolved in a solvent (usually water), allowing the solution to conduct electricity. The ions in the solution can move freely, which makes the solution conductive: **Examples: Strong Electrolyte Solutions:** Solutions of sodium chloride (NaCl), hydrochloric acid (HCl), or potassium hydroxide (KOH).

■ Non-electrolyte Solution:

Definition: A non-electrolyte solution is a solution where the solute does not dissociate into ions and, as a result, does not conduct electricity. The molecules remain intact in the solution.

- **Examples:** Solutions of sugar (sucrose), ethanol, or urea in water.

■ **Suspension:** A suspension is a heterogeneous mixture where solid particles are dispersed in a liquid or gas, but these particles are large enough to be visible to the naked eye or under an optical microscope. The particles in a suspension tend to settle at the bottom after a certain time if left undisturbed due to gravity. A common example is mud in water.

- **Example:** sand in water
- **Particle size:** larger than 1 μm (micrometer)

- **Tendency to settle:** yes, particles settle at the bottom over time
- **Colloid:** A colloid is a mixture where microscopic particles, smaller than those in a suspension but larger than those in a solution, are dispersed in a liquid, gas, or solid. Colloidal particles are generally between 1 nm (nanometer) and 1 μm (micrometer) in diameter. Unlike suspensions, colloids are stable, and particles do not settle easily. A typical effect observed in colloids is the Tyndall effect, where light is scattered by the particles.
 - **Example:** milk, jelly
 - **Particle size:** between 1 nm and 1 μm
 - **Tendency to settle:** no, particles remain suspended
- **Homogeneous Mixture:**

Definition: A homogeneous mixture is a mixture where the components are evenly distributed and cannot be distinguished from one another. The composition is uniform throughout : **Examples:** Vinegar, air.
- **Heterogeneous Mixture:**

Definition: A heterogeneous mixture is a mixture where the components are not uniformly distributed, and different parts of the mixture can be observed or separated physically: **Examples:** Salad, oil and water.

 - The term **miscible** refers to the ability of two liquid substances to completely mix with each other to form a homogeneous solution without separating into two distinct phases. Miscible liquids, such as alcohol and water, mix in any proportion, regardless of the quantity of each, to create a single uniform phase. In contrast, **non-miscible** liquids (such as water and oil) do not mix and form two separate layers.

I.1 Generalities on Separation Methods

Separation techniques rely on differences in the physical or chemical properties of the components in a mixture. The most commonly exploited properties are:

- Particle size
- Density
- Melting point
- Solubility
- Polarity
- Volatility
- Affinity for a solvent or adsorbent

■ Common Separation Methods:

- Filtration
- Centrifugation
- Decantation
- Distillation
- Liquid-liquid extraction
- Chromatography
- Evaporation
- Crystallization

Each method is chosen based on the characteristics of the mixture to be separated and the desired result (purification, isolation, concentration).

I.1 Case of a Solid-Liquid Mixture:

(a) Filtration:

Filtration is one of the simplest techniques to separate a solid from a liquid. It is based on the difference in particle size.

- **Principle:** A filter (filter paper, membrane, sieve) is used to retain solid particles while allowing the liquid (filtrate) to pass through.
- **Application:** Separation of mud from water, extraction of a precipitate formed during a chemical reaction.

Generally there are two modes of filtration, simple filtration and vacuum filtration:

(a) Simple filtration:

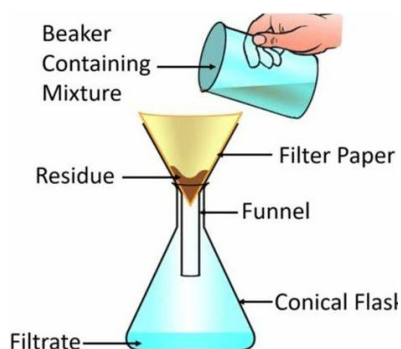
Simple Filtration is a separation technique used to remove solid particles from a liquid by passing the mixture through a filter. The process is based on the difference in particle sizes, where larger solid particles are retained by the filter while the liquid phase (filtrate) passes through.

■ Principles of Simple Filtration

- **Required Equipment:** A funnel, filter paper, a beaker or Erlenmeyer flask, and a mixture containing solid and liquid phases.
- **Procedure:**
 - ✓ The solid-liquid mixture is poured into a funnel lined with filter paper.
 - ✓ The liquid passes through the filter paper, collecting as the **filtrate** in the container below.
 - ✓ The solid particles remain on the filter paper, forming the **residue**.

■ Applications

- ✓ Separation of precipitates in chemical reactions.
- ✓ Clarification of liquids by removing solid impurities.
- ✓ Purification of solutions in laboratory and industrial processes.



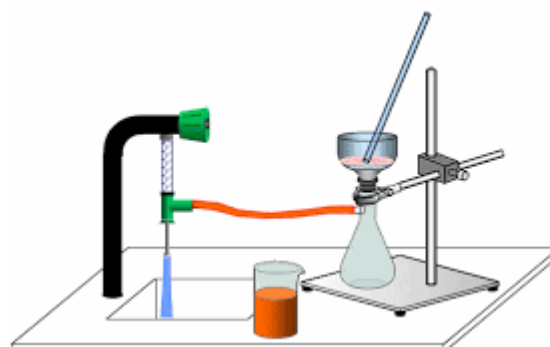
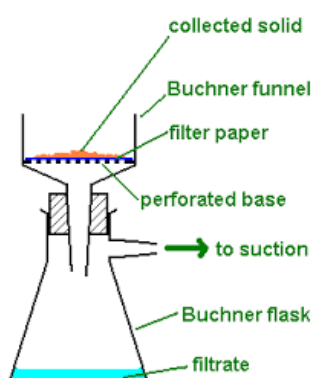
Simple filtration

(b) Büchner Filtration

Büchner filtration is a vacuum filtration method used to quickly separate solids and liquids in a mixture. It employs a Büchner funnel and a vacuum pump to accelerate the filtration process. This technique is widely used in chemistry to isolate precipitates or purify products.



Büchner funnel



Büchner Filtration

■ Required Equipment

- Büchner funnel (ceramic or plastic)
- Filter paper suited to the Büchner funnel
- Filtration flask (or vacuum flask)
- Vacuum pump
- Rubber hose
- Rubber ring or gasket to ensure a tight seal
- Mixture to be filtered

■ Operating Principle

Büchner filtration relies on applying negative pressure (vacuum) below the funnel to speed up the filtration. The liquid is quickly drawn through the filter paper, while the solids are retained on the paper.

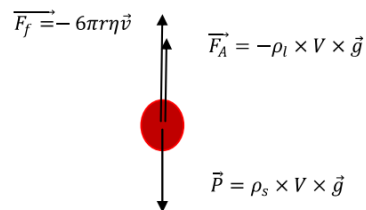
(b) Sedimentation

- **Definition:** The process by which solid particles settle to the bottom of a fluid.
- **Applications:** Water treatment, chemical, pharmaceutical, and food industries, soil analysis, etc.
- **Types of Sedimentation:**
 - **Free Sedimentation:** Particles fall without interacting with each other.
 - **Hindered Sedimentation:** Particles interact due to high concentration.

■ Theoretical Principles

- **Stokes' Law:** Describes the settling velocity of spherical particles in a viscous fluid:

$$v = \frac{2(\rho_p - \rho_f)gr^2}{9\eta}$$



where:

- ✓ V : settling velocity of the particle
- ✓ ρ_p : density of the particle
- ✓ ρ_f : density of the fluid
- ✓ g : acceleration due to gravity
- ✓ r : radius of the particle
- ✓ η : dynamic viscosity of the fluid

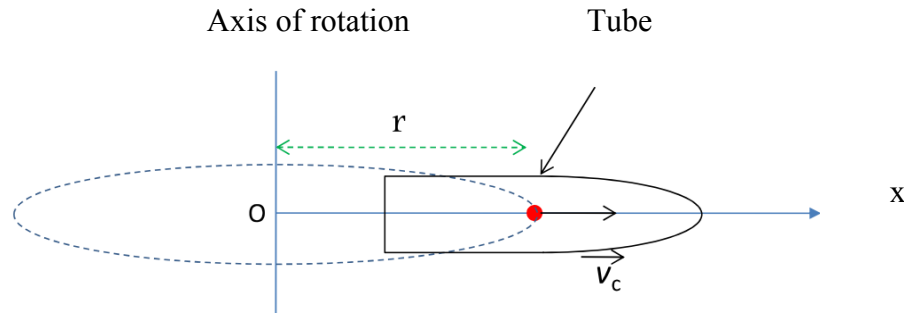
■ Factors Affecting Sedimentation:

- ✓ Particle size and density
- ✓ Fluid viscosity and density
- ✓ Temperature (it affects viscosity)
- ✓ Particle shape (deviations from sphericity)

(c) Centrifugation:

Centrifugation is a faster and more efficient technique than filtration for separating solid and liquid phases.

- ✓ **Principle:** Centrifugation relies on centrifugal force to accelerate the sedimentation of solid particles based on their density.
- ✓ **Application:** Separation of blood components (plasma and cells), separation of solid and liquid phases in colloidal solutions.



$$v_c = \frac{2(\rho_p - \rho_f)\gamma d^2}{9\eta} \quad \text{and} \quad \gamma = r \times \omega^2$$

Where :

γ : Acceleration [m/s^2]

ω : Centrifuge rotation speed; [rad/s]

(r) : Radius; m



Centrifuge

Centrifuge tube

(d) Évaporation

Evaporation is a physical process used to separate a volatile solvent from a mixture by transforming it into vapor through heating. This method is especially useful for recovering the solute or concentrating a solution by increasing the temperature to allow the solvent to evaporate. For example, to isolate salt from saltwater, the solution is heated until the water fully evaporates, leaving salt as a solid residue.

(e) Drying

Drying is a process that removes water or any other liquid solvent from a wet solid. This method can be performed in open air, in an oven, or using a specialized dryer. Drying is often used to treat solid products after extraction, filtration, or washing, to return them to a dry state. This process is commonly used in food, pharmaceutical, and chemical industries.

(f) Adsorption

Adsorption is a process where molecules of a liquid or gas adhere to the surface of a solid (known as an adsorbent). This process is mainly used to remove impurities or specific components from a liquid or gas mixture. A common example is the use of activated carbon to adsorb organic substances and certain impurities in drinking water or to purify air. Adsorption is widely used in water treatment, pollution control, and in certain chemical processes.

I.2 Case of Two Immiscible Liquids:

1. Decantation:

Decantation is a method used to separate two immiscible liquids that do not mix (e.g., oil and water).

- **Principle:** The two liquids separate based on their density, with the denser liquid settling at the bottom and the less dense one remaining on top. They can then be separated using a separatory funnel.
- **Application:** Separation of water and oil, extraction of an aqueous phase and an organic solvent.