ALGERIAN DEMOCRATIC AND PEOPLE'S REPUBLIC

Ministry of Higher Education and Scientific Research

University of Mostaganem Abdelhamid Ibn Badis

Faculty of Science and Technology

Department of Process Engineering

Course Handout

Unit Operations

Intended for 3rd-year undergraduate students in Process Engineering

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Academic Year: 2025-2026

NATIONAL PROGRAMME 2025 – 2026 (3rd update)

Semestre :6
Unité d'enseignement : UEF 3.2.1
Matière1: Opérations unitaires
VHS: 67h30 (Cours: 3h00, TD: 1h30)
Crédits : 6
Coefficient : 3

Objectifs de l'enseignement:

Connaître les principales opérations unitaires et comprendre les schémas des procédés des différentes industries du génie des procédés (chimiques, électrochimiques, agroalimentaires, pharmaceutiques, …, etc.); Ecrire et contrôler les bilans matières de ces processus.

Connaissances préalables recommandées:

Thermodynamique; Equations différentielles; Phénomènes de transfert.

Contenu de la matière:

Chapitre 1 : (1 semaine)

Généralités sur les opérations unitaires: Absorption ; Extraction ; Adsorption ; Distillation, etc…

Chapitre 2 : (3 semaines)

Absorption : Equilibre liquide-gaz ; Absorption isotherme, Bilans de matière; Concept d'étage théorique ; Méthode de Mac Cabe et Thièle, notions de contacteurs (colonnes garnies et à plateaux), hydrodynamique des écoulements.

Chapitre 3 : (4 semaines)

Extraction Liquide – Liquide : Introduction ; définition (solvant, soluté, diluant), Diagramme d'équilibre ; Extraction à un seul étage; extraction multiétages : Méthode graphique de Mac Cabe et Thièle, nombre de plateaux théoriques

Chapitre 4 : (3 semaines)

Extraction liquide-solide (Lixiviation) : Equilibre solide-liquide ; Diagramme de Janeck : Détermination du nombre d'étages théoriques, cas de l'extraction à contre-courant et à courants croisés.

Chapitre 5 : (4 semaines)

Distillation : Distillation d'un mélange binaire ; Distillation en mode discontinu, continu ; Calcul de l'efficacité d'une colonne de rectification (Méthodes graphiques de Mac Cabe et Thièle et de Ponchon et Savarit).

Mode d'évaluation:

Contrôle continu: 40%, Examen: 60%.

Références bibliographiques:

Robert E. Treybal,«Mass transfer operations», MC Graw Hill.

MC Cabe et Smith,« Chemical engineering operations», MC Graw Hill.

3. COULSON J.M., J.F RICHARDSON, J.R BACKHURST and J.H. HARKER, "Chemical Engineering", volume two, Fifth edition, 2002.

Résumé

Ce polycopié présente les principes fondamentaux et les méthodes de calcul des principales opérations unitaires de transfert de matière utilisées en génie des procédés. Il traite successivement de l'absorption gaz-liquide, de l'extraction liquide-liquide, de l'extraction liquide-solide (lixiviation) et de la distillation. Les notions d'équilibre entre phases, les bilans de matière, les concepts d'étages théoriques ainsi que les principales méthodes graphiques de dimensionnement et d'analyse, notamment celles de McCabe-Thiele, Ponchon-Savarit et Janeck, sont développées. Ce cours permet aux étudiants d'acquérir les bases nécessaires à l'analyse, à la conception et à l'exploitation des équipements industriels impliqués dans les procédés de séparation.

Mots-clés : Opérations unitaires, Génie des procédés, Distillation, Absorption, Extraction liquide-liquide, Transfert de matière, Procédés de séparation.

Abstract

This course manual presents the fundamental principles and calculation methods of the main mass-transfer unit operations used in chemical and process engineering. It covers gas-liquid absorption, liquid-liquid extraction, liquid-solid extraction (leaching), and distillation. Phase equilibrium, material balances, theoretical stage concepts, and the main graphical design and analysis methods, including McCabe-Thiele, Ponchon-Savarit, and Janecke approaches, are discussed. This course provides students with the essential knowledge required for the analysis, design, and operation of industrial separation processes and equipment.

Keywords : Unit Operations, Process Engineering, Distillation, Absorption, Liquid-Liquid Extraction, Mass Transfer, Separation Processes.

ملخص

يتناول هذا المطبوعة التعليمية المبادئ الأساسية وطرق الحساب الخاصة بأهم العمليات الوحدوية المعتمدة على انتقال المادة في هندسة الطرائق. ويشمل دراسة الامتصاص غاز-سائل، والاستخلاص سائل-سائل، والاستخلاص سائل-صلب (الترشيح أو الإذابة الاستخلاصية)، والتقطير. كما يعرض مفاهيم توازن الأطوار، وموازين المادة، ومفهوم المراحل النظرية، إضافة إلى أهم الطرق البيانية المستخدمة في التحليل والتصميم مثل طريقة ماكابي وثيل، وطريقة بونشون وسافاري، ومخطط جانيك. يهدف هذا المقرر إلى تزويد الطلبة بالمعارف الأساسية اللازمة لتحليل وتصميم وتشغيل معدات وعمليات الفصل الصناعية.

الكلمات المفتاحية : العمليات الوحدوية، هندسة العمليات، التقطير، الامتصاص، الاستخلاص سائل-سائل، انتقال المادة، عمليات الفصل.

Foreword

This course handout is intended for third-year students in the Bachelor's program in Process Engineering (LMD). Its objective is to provide a solid and structured foundation in the field of unit operations, which constitute a fundamental pillar in the understanding and design of industrial processes.

Unit operations encompass all physical techniques for transfer matter, such as the transfer of mass, heat, and momentum. Mastering these concepts is essential for any engineer working in diverse sectors such as the chemical, petrochemical, food processing, or environmental industries.

This document has been prepared with educational clarity in mind. It offers a step-by-step introduction to fundamental concepts, accompanied by application examples and exercises that allow students to consolidate their knowledge and develop their scientific reasoning.

The main objective of this handout is to help students understand the phenomena involved in unit operations, master the associated calculation tools, and be able to apply this knowledge to real-world situations.

This handout is divided into five chapters as follows: the first chapter provides a general introduction and covers the main types of unit operations.

The second chapter is devoted to the liquid-liquid extraction unit operation, covering single-stage extraction, co-current and counter-current multi-stage extraction, and the determination of the number of theoretical plates.

The third chapter presents liquid-solid extraction; the objective of this chapter is to understand: the different types of solid-liquid extraction, the concept of a transfer stage, mass balance, solid-liquid equilibrium, and the Janeck diagram.

The fourth chapter focuses on the phenomena of absorption and desorption. It begins with the influence of temperature and pressure, covering mass balance, liquid-vapor equilibrium, the MacCabe and Thièle methods, concepts of contactors, and flow hydrodynamics.

The final chapter is devoted to distillation: distillation of a binary mixture, batch and continuous distillation, and calculation of the efficiency of a rectification column.

All chapters share a common structure: definition and principles, application examples, and various types of equipment.

This material does not replace reference works but serves as a supplementary guide tailored to the curriculum and students' needs. Furthermore, this handout is intended solely as a supplement to the course and in no way replaces the student's attendance in class.

Finally, any comments or suggestions aimed at improving this work are welcome.

Author

For any suggestions, email: karima.menad@univ-mosta.dz

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Chapter I.

General Information on Unit Operations

A. Introduction to unit operations 'UO'

The term "operation" encompasses all separation processes or techniques, such as distillation, extraction, drying, filtration, and crystallization.

These processes are called unit operations because they are based on the same physical principles and involve simple transformations.

Their operation is based on the following fundamental principle:

1) Intimate contact between phases;

2) Mass transfer (MT) and/or heat transfer (HT) and/or momentum transfer during this contact period;

3) Separation of the constituents (solutes) of the resulting mixture.

- **Classification**

Based on the second step of the principle, the various UO can be classified as follows:

1. UO with or without TC
Example: liquid-liquid extraction and drying

2. UO based on fluid mechanics
Example: centrifugation: separation of a mixture of two liquid phases or a solid phase suspended in a liquid phase, through the action of centrifugal force.

3. UO based on mechanical principles
Example: sieving: separation of solid particles by size using a sieve.

4. UO with or without TM
Example: Filtration: is a separation process used to remove solid particles from a fluid (liquid or gas) by passing it through a porous medium called a filter, which retains the solids while allowing the fluid to pass through. It is a separation process without mass transfer.

These examples of separation with matter transfer are distinguished:

- **Liquid-Liquid Extraction (Chapter II)**
- **Solid-liquid extraction (Chapter III)**
- **Absorption, desorption (Chapter IV)**
- **Distillation (Chapter V)**

B. The three main objectives of OPUs

1. Purification of the diluent (the solute is an impurity)

This operation aims to remove unwanted solute impurities from the diluent in order to obtain a purer main component. Common separation methods include distillation, filtration, extraction, and adsorption, depending on the nature of the impurity.

Example 1: Liquid-liquid extraction: this involves recovering a solute dissolved in a first liquid phase (diluent) into a second liquid phase (solvent) such that the solute is more soluble in the solvent than in the initial solution (diluent)

(Unit 200; Zone 5 "base oil production area"; Arzew oil refinery: deasphalting—removal of asphalt (solute) contained in a grade of oil (diluent) using propane (solvent))

Example 2: Absorption: a process based on the transfer of a solute from a gas to a liquid. (Decarbonation: reduction of the amount of CO2 in natural gas through the use of MEA as a liquid). We can also mention adsorption: the binding of molecules to the surface of solids.

2. Solute recovery (the solute in an important substance)

The recovery of a valuable solute is carried out using appropriate unit operations such as filtration, extraction solid liquid, adsorption, crystallization, or membrane separation.

Example: Solid-liquid extraction: the transfer of a solute from a solid to a liquid.

(Coffee preparation: coffee, which contains aromatic compounds—the "solute"—comes into contact with water—the "solvent"—to produce drinkable coffee)

3. The behavior of multiple solutes

The third objective of unit operations is to study the behavior of multiple solutes, including their interactions and separation during processing.

This involves recovering the components of a mixture.

Example: Distillation: the separation of multiple solutes from a homogeneous liquid phase based on their boiling points.

Unit operations are used in many industries, including chemical, petroleum, pharmaceutical, and food processing. They enable the production, purification, and separation of products. Industrial plants combine multiple unit

(Unit 100; Zone 5; Arzew oil refinery: distillation of atmospheric-reduced crude (BRA) from Zone 4. This distillation yields three grades of oil based on their different boiling points.

Chapter II.
Liquid-Liquid Extraction

1. Partition coefficient;

2. Selectivity;

3. Different types of diagrams;

4. Equipment used in continuous and batch processes;

5. Partially soluble solvent: multistage co-current and counter-current extraction (Ternary diagram);

6. Insoluble solvent: multi-stage co-current and counter-current extraction (construction Mac Cabe and Thièle);

7. Apparatus;

8. Application Exercises.

II.1. Principles and Definitions

Consider a solution in which solute **A** is dissolved in diluent **D**. When separating A from D by distillation is technically difficult or economically impractical, an alternative method is required. In such cases, a third component, the extraction solvent **S**, is introduced. This solvent is chosen because it has a high affinity for solute A, but is only slightly miscible with diluent D. This allows solute A to be extracted selectively. The diagram of this extraction is shown in Fig II.1.

In fact, since total insolubility does not exist, we always recover:

1- A raffinate consisting of D depleted in A but containing a small amount of S.

2- An extract rich in S containing A and a small amount of D.

This process is athermic and requires very little energy (stirring). It is a superior alternative to distillation in cases of difficult separation. It is sometimes combined with distillation of the extract, allowing for the recovery of the solvent on the one hand, and the solute with the desired purity on the other.

Fig. II.1. Schematic diagram of a liquid-liquid extraction process.

Since there is no accumulation of material in the extraction apparatus, the mass entering is equal to the mass leaving.

This process can be carried out batchwise (e.g., using a laboratory separatory funnel) or continuously (using mixing separators, extraction columns, etc.).

Note: If A crystallizes easily, an alternative process is fractional crystallization, which involves precipitating it from the mixture by lowering the temperature.

II.2. Liquid-liquid extraction terminology

- Solute: the substance to be extracted;
- Diluent: liquid containing the solutes;
- Feed solution (the feed): mixture of solute and diluent;
- Solvent: liquid used to extract the solutes;

- Extract: phase resulting from the operation containing the extracted solutes. This phase is rich in solvent;
- Raffinate: residual phase depleted of solute. This phase is rich in diluent;
- Heavy phase: the phase with the highest density;
- Light phase: the phase with the lowest density.

II.3. Some major processes using extraction

Here are some examples using the liquid-liquid extraction process.

- separation of rare earth elements (site in La Rochelle).

(is one of the leading groups in the French chemical industry specializing in fine chemicals)

- Separation of acetic acid from water using ethyl ether or ethyl acetate.
- Recovery of aromatics from aliphatics using the UDEX process.

(Aromatic: Benzene C_6H_6, aliphatic: ethane C_2H_6: an organic compound with at least one C-H bond that may be linear; UDEX: universal-dow-extraction, the first industrial process for the extraction of aromatics to become available).

- Kerosene refining.
- Long-chain fatty acids can be separated from vegetable oils by vacuum distillation, but more economically by liquid extraction with liquid propane.
- Tantalum and niobium can be separated by a long and delicate fractional crystallization process, but more easily by liquid extraction of a hydrofluoric acid solution using methyl isobutyl ketone (MIBK).
- Many unstable or heat-sensitive pharmaceutical products, such as penicillin, are obtained from mixtures so complex that only solvent extraction meets the requirements of the process.

II.4. The distribution coefficient and distribution curve

Solvent extraction requires two steps:

1- Bringing the liquid containing the solute into as intimate contact as possible with the chosen solvent. This contact is maintained for as long as necessary to achieve equilibrium between the two phases.

2- The mechanical separation of the two phases, which yields:

– The solution, rich in solvent; it is called the extract;

– The residue, which is a second liquid layer rich in solvent, is called the raffinate.

The distribution or partition coefficient "k" represents the ratio of the total solute concentration in the extract to the total concentration of solute in the raffinate at equilibrium.

$k = C_A$ in the extract / C_A in the raffinate

Or: $k = y_A / x_A$ such that:

x_A: Fraction (by mass or molar) of the solute in the raffinate at equilibrium.

y_A: Fraction (by mass or molar) of the solute in the extract at equilibrium.

The curve $y_A = f(x_A)$ is an equilibrium curve for solute between the two phases. It is called the distribution (or partition) curve (see Fig. II.2).

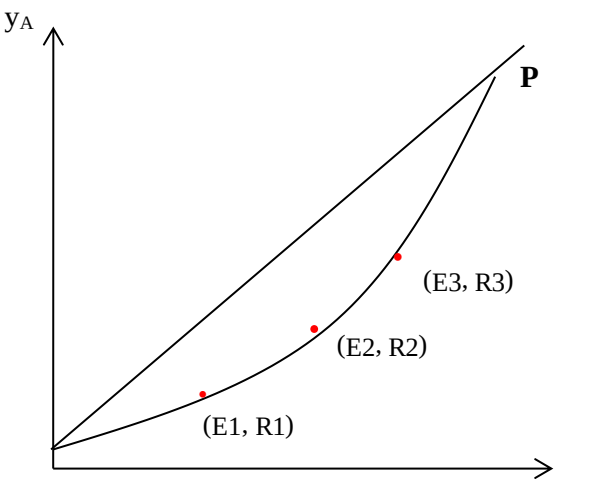

Fig. II.2. Distribution curve.

II.5. Selecting a temperature and a solvent

II.5.1. Choosing a temperature:

It is often advantageous to operate at the lowest possible temperature, as solubility ranges decrease with temperature.

II.5.2. Choosing a solvent:

Obviously, the solvent must be chosen:

Which selectively extracts the solute to be recovered. *The solute is more soluble in the solvent than in the solution in which it is initially present;*

- Which may be insoluble or slightly soluble in the diluent;
- Which has high solvent power, to minimize the amount of solvent used;
- Which has a density sufficiently different from that of the solution to be purified, so that separation by decantation between the two phases after extraction is easy;
- Finally, one that is not expensive and does not pose any prohibitive risks in terms of toxicity, flammability, or corrosion, and that separates easily from the solute it has extracted.

II.6. Graphical representation

The simplest system encountered in liquid-liquid extraction is the ternary system, where a single solute is distributed between two liquids that are slightly or not at all miscible.

II.6.1. Graphical representation terminology

A: solute;

D: diluent;

S: solvent;

L_o: mass of pure solvent;

R_o: mass of the A + B mixture (the feed or charge);

R_i: mass of the raffinate after extraction;

E_i: mass of the extract after extraction;

it can be expressed as a mass flow rate (if the process is continuous)

x: mass fraction in Ro nd Ri;

y: mass fraction in Lo and Ei;

z: mass or molar fraction in the mixture;

M: mixing point;

M_{total}: total mass of the mixture (before separation into Ri and Ei).

Graphical methods play an essential role in the analysis and design of liquid–liquid extraction operations. For ternary systems, equilibrium data are commonly represented on triangular diagrams, where each vertex corresponds to a pure component and every point inside the triangle represents a specific mixture composition. The **feed (F)** denotes the initial solution containing the solute to be extracted, whereas the **solvent (S)** represents the extracting agent introduced into the system. The line joining F and S is known as the **mixing line**, and any point on this line corresponds to a possible mixture of feed and solvent. The resulting composition after mixing is called the **mixture point (M)**. When the mixture enters the two-phase region, it separates into an **extract phase (E)**, rich in solvent and extracted solute, and a **raffinate phase (R)**, rich in the original diluent. The equilibrium compositions of these two phases are connected by **tie lines**, which provide valuable information on phase distribution. The boundary separating the homogeneous and heterogeneous regions is called the **binodal curve** or **solubility curve**.

II.6.2 Reminder on triangular diagrams

We know how to represent a **ternary mixture** on a triangular diagram, which can be equilateral or isosceles-rectangular.

- Each vertex represents a pure substance;
- Each vertex represents a pure component of the ternary mixture. The lower-right vertex represents the solvent; the top vertex represents the solute. The last vertex (bottom left) obviously represents the diluent;
- Each side represents a binary mixture;
- Each point in the middle represents a ternary mixture;
- The percentage of concentrations increases counterclockwise;

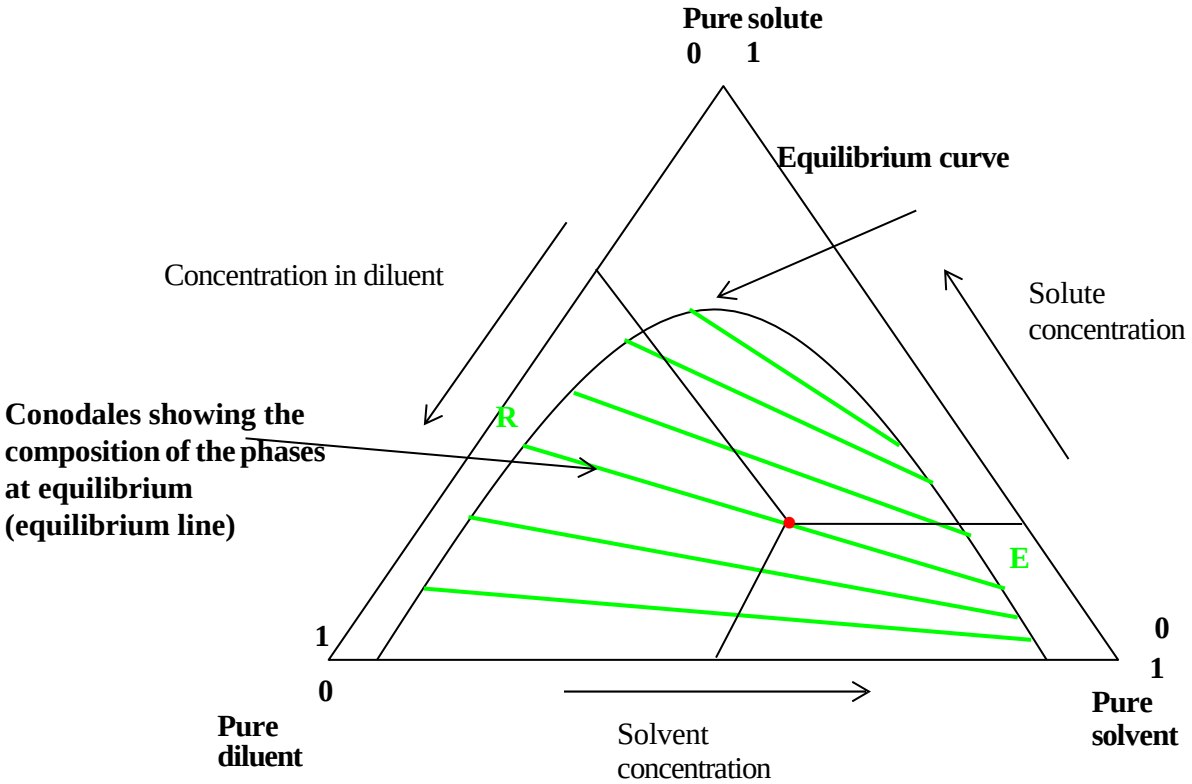

Fig. II.3. The equilateral triangle diagram.

- R and E are placed respectively:

  -R, near line AD because D has been purified of A; R represents the raffinate;

  -E, near line AS because S has extracted A but remains rich in S; E represents the extract.

In this diagram, the curve drawn inside the triangle represents the miscibility limit of the two liquid phases. Below the curve, the mixture represented by point M is heterogeneous and separates into two liquid phases. Above the curve, the mixture represented by point M would be homogeneous.

II.6.3 Reading the equilateral triangular diagram

A. Mixture point M

Simply draw lines from point M that are parallel to the bases and opposite the vertices, and read the labels on the axes formed by the bases (see fig. II. 4).

$Ma + Md + Ms = AD = DS = SA$

$Ad + Ds + Sa = AD = DS = SA$

M represents the mixture with

composition: $z_A = Sa$

$z_D = Ad$

$z_S = Ds$

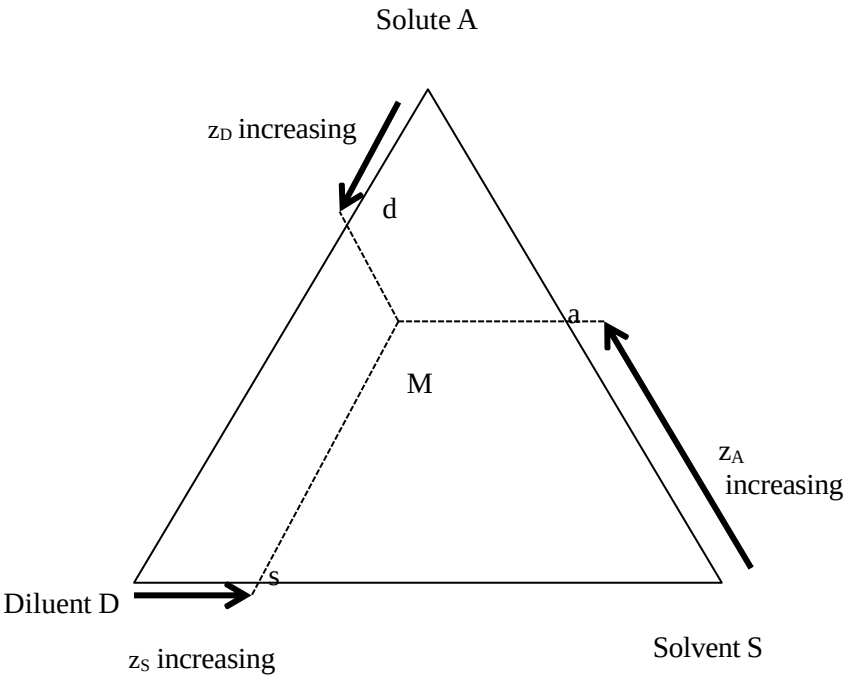

Fig II.4. representation of point M.

B. Equilibrium line and curve

To determine the equilibrium lines, it is necessary to determine the distribution curve experimentally, and the fractions of the three components—whether in the raffinate (x_A, x_D, x_S) or in the extract (y_A, y_D, y_S)—are determined by the same experiment. The procedures for determining the equilibrium line and curve are shown in the following figure (case of $k < 1$, distribution curve above the first diagonal):

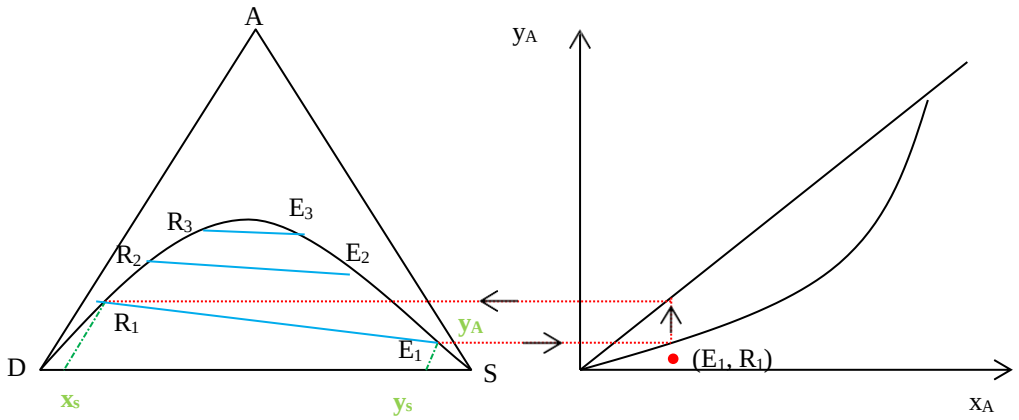

Fig. II.5. Determination of the equilibrium line and curve.

II.6.4. Reading the isosceles-rectangular triangular diagram

The representation of the mixing point M is shown in the following figure. M represents the mixture with composition:

z_A = Dd

z_S = Ds

z_D = 1 – z_A - z_S = Ma

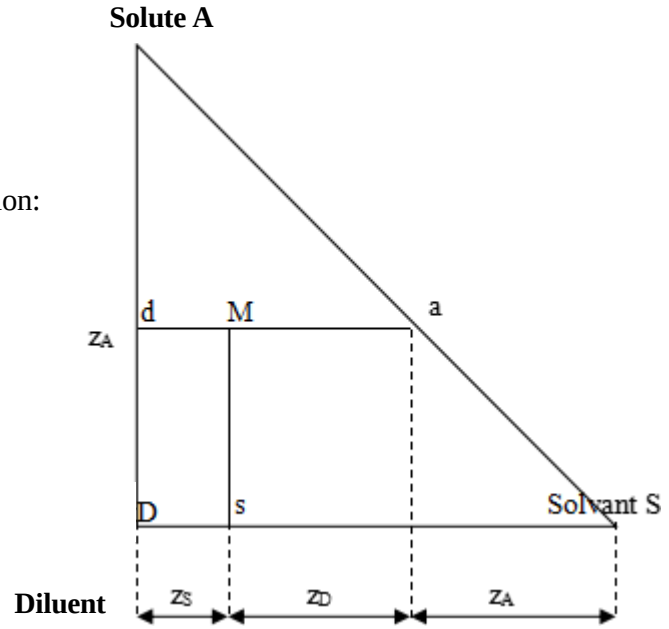

Fig II.6. Representation of point M on isosceles-rectangular triangular diagram.

Total immiscibility of D and S: The solubility isotherm (miscibility curve) coincides with the sides AD and AS of the triangle.

In this case, we always have:

x_S = 0 in the raffinate

y_D = 0 in the extract

Here is such a diagram in triangular coordinates:

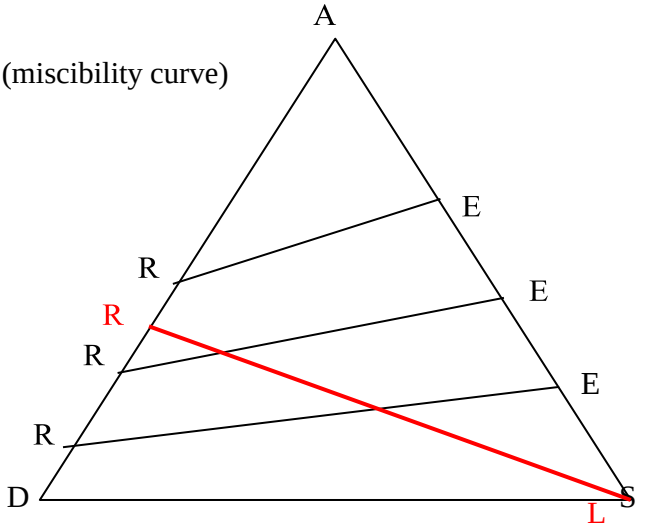

Fig II.7. Triangular diagram in the case of total immiscibility of diluent and solvent.

II.7. Liquid-liquid extraction processes (diluent and solvent are poorly miscible with each other)

II.7.1. Single-stage extraction (single-stage extraction)

This procedure is performed batch wise in the laboratory using separatory funnels. It can, however, be carried out continuously, and in both cases the process flow can be represented by the following figure.

A. Overall mass balance:

Input = Output

$R_0 + L_0 = R_1 + E_1 = M_{total}$ …. **(1)**

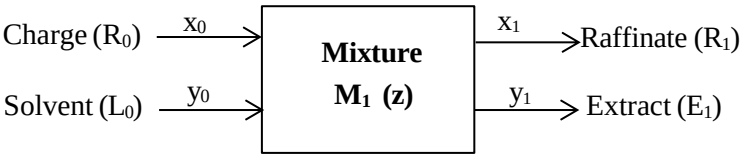

Fig. II.8. Schematic of single-stage extraction.

B. Partial mass balance for each component:

- Solute A

$R_0\, x_{0A} + L_0\, y_{0A} = R\, x_{1A} + E\, y_{1A} = z_{1A}\, M_{total}$ …. **(2)** where $M_{total} = R_0 + L_0$

Therefore, $\mathbf{z_{1A} = R_0\, x_{0A} / (R_0 + L_0)}$ ….. **(3)**

- Diluent D

$R_0\, x_{0D} + L y_{0D} = R x_{1D} + E y_{1D} = z_{1D}\, M_{total}$, so $\mathbf{z_D = R_0\, x_{0D} / (R_0 + L_0)}$ ….. **(4)**

- Solvent S

$R_0\, x_{0S} + L y_{0S} = R x_{1S} + E y_{1S} = z_{1S}\, M_{totale}$ donc $\mathbf{z_S = L_0\, y_{0S} / R_0 + L_0}$ ….. **(5)**

C. Graphical representation of the mixture point M_1:

The value of z_{1A} thus allows us to locate point M_1 (the parallel line DS corresponding to the composition z_{1A} intersects segment R_0L_0 at M_1) such that R_0 and L_0 are determined by the method.

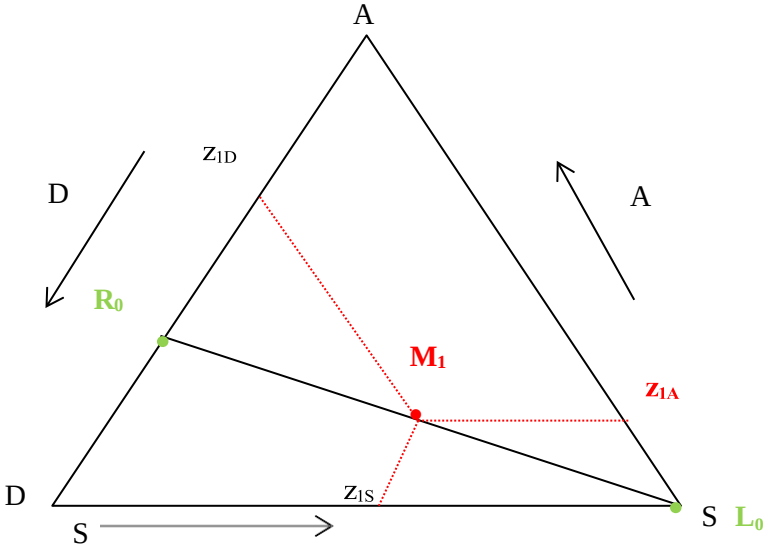

Fig II.9. Graphical representation of the mixture point M_1.

D. Compositions and quantities of the streams after extraction

Combining (1) and (2) yields: (R_0 and L_0 are determined by the process)

$R_1 = M_{totale} - E_1$ et $R_1 x_{1A} + E_1 y_{1A} = z_{1A} M_{totale}$ **so** $(M_{totale} - E_1) x_{1A} + E y_{1A} = z_{1A} M_{totale}$

$E_1 (y_{1A} - x_{1A}) = M_{totale} (z_{1A} - x_{1A})$ so $\mathbf{E_1 = M_{totale} (z_{1A} - x_{1A}) / (y_{1A} - x_{1A})}$ **. . . . . (6)**

$$\mathbf{R_1 = M_{totale} - E_1} \text{.....(7)}$$

$$\mathbf{R_1 = M_{totale} (z_{1A} - y_{1A}) / (x_{1A} - y_{1A})} \text{ . . . (8)}$$

To determine the compositions $\mathbf{x_{1A}}$ **and** $\mathbf{y_{1A}}$**,** we refer to the diagram on the figure II. 10. We use the distribution curve, which allows us to plot the equilibrium line passing through point M1 by interpolation, and read the corresponding fractions (see diagram).

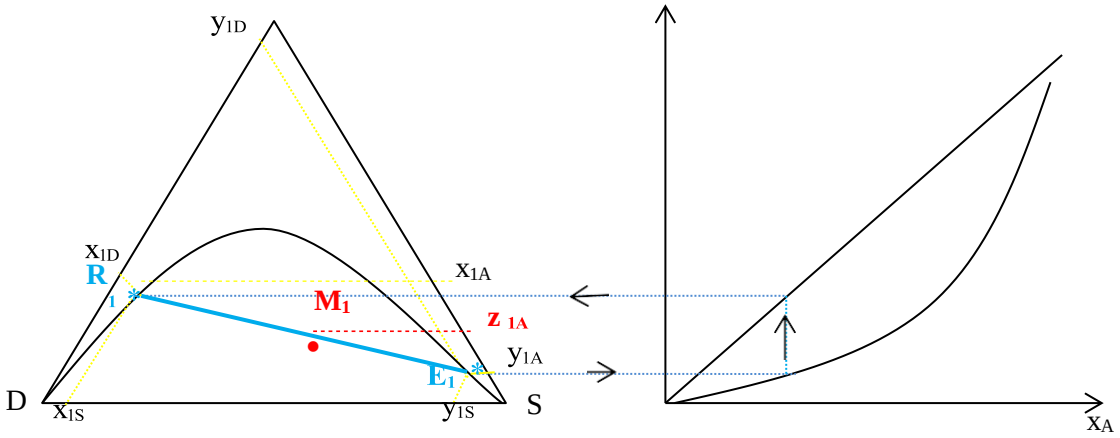

Fig II.10. Graphical determination of compositions.

E. Solvent selectivity using the triangular diagram

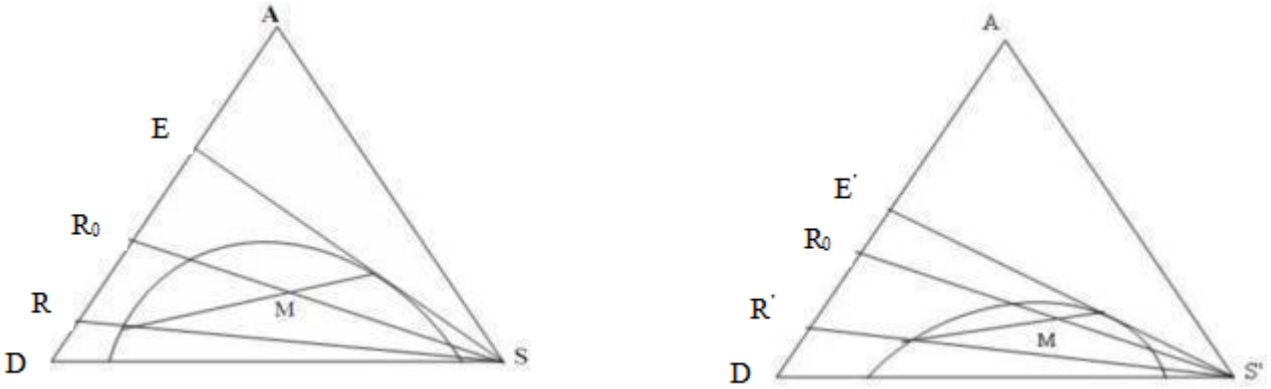

Fig. II. 11. Solvent selectivity.

A solvent (S) is selective with respect to a binary mixture of (A) and (D) if it dissolves solute (A) preferentially over another solvent (S'). Consider the following figure, where solvents (S) and (S') are used to separate the components of a binary mixture represented by point (R_0). The phases obtained after solvent separation are (E) or (E') for the extract and (R) or (R')

for the raffinates. The figure below shows that the selectivity of (S) is better than that of (S'), since the representative points (E) and (R) are farther from ($_{R0}$) than (E') and (R').

- Selectivity can be defined in a similar way to relative volatility in distillation:

$$\alpha' = \frac{y_A}{x_A} \times \frac{x_B}{y_B} \qquad \alpha' = K_A / K_D$$

The separation becomes more effective as α′ increases above 1; it is possible if α' = 1.

II. 7. 2. Multi-stage co-current extraction

This process is an extension of single-stage extraction; the raffinate R_1, R_2… Rn is brought into contact with fresh solvent at each stage.

It requires a large amount of solvent and is carried out in mixer-settlers that make up the various stages. It is mainly used in batch operations in laboratories or small industrial plants.

The schematic diagram is illustrated in figure II. 12:

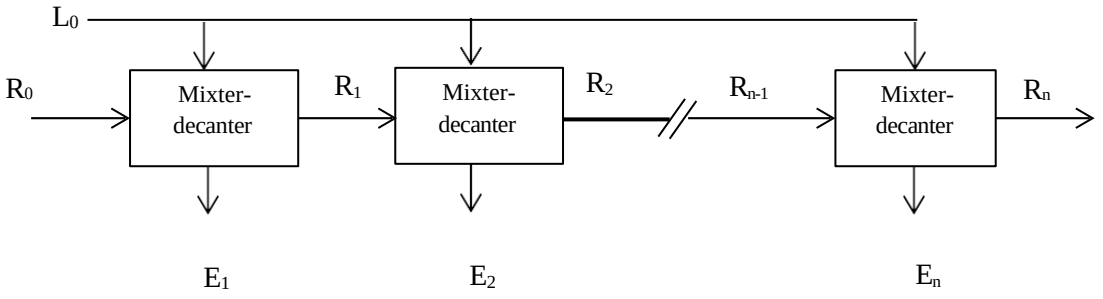

Fig. II.12. Schematic of a co-current multiple-contact extraction.

It can supply each tray with a quantity of fresh solvent, which does not necessarily have to be identical or pure.

A. Material balance:

For the first floor:

$$R_0 + L_0 = R_1 + E_1$$

$$R_0 x_0 + L_0 y_0 = R_1 x_1 + E_1 y_1 = M_{t1} . z_1$$

For the second stage:

$$R_1 + L_1 = R_2 + E_2$$

$$R_1 x_1 + L_0 y_0 = R_2 x_2 + E_2 y_2 = M_{t2} . z_2$$

For the n^{th} stage:

$$R_{n-1} + L_{n-1} = R_n + E_n$$

$$R_{n-1} x_{n-1} + L_0 y_0 = R_n x_n + E_n y_n = M_{tn} . z_n$$

B. Mixing point, fraction and mass of extract and raffinate phases

The calculation procedures and graphical presentation of this extraction are analogous to those described in the single-stage extraction. The triangular diagram is used to determine the mixing point of each stage as well as the fractions of the extract and the raffinate after each extraction.

The same procedure is used to determine the various values for the second stage: M_2 is obtained by connecting R_1 with L_0 (where L_0 is the quantity and remains the same) and with the intersection of the segment parallel to DS, which corresponds to the value z_{2A} (z_{2A} is determined by material balance—Equation 3); then, by interpolation, R_2 and E_2 are obtained, and the fractions are read from the graph, and the quantities are calculated using Equations 6 and 7.

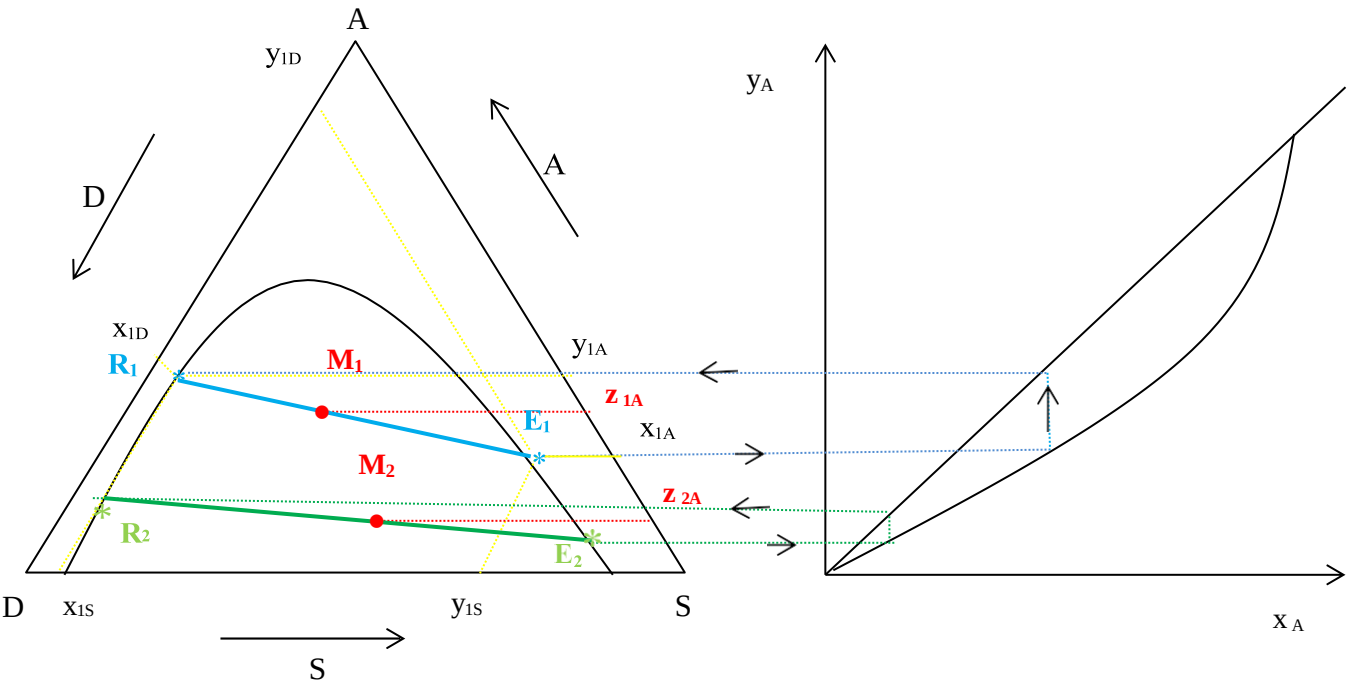

Fig II.13. Graphical determination of compositions of mixing point, extract and raffinate phases for two floors.

II. 7. 3. Multi-stage countercurrent extraction

This process involves circulating the solution to be extracted, R_0, and the solvent, L_0, in opposite directions through various stages of a column, in which the raffinate, R_n, becomes depleted in solute while the extract, E_n, becomes enriched in solute. This process is widely used industrially and consumes less solvent than the previous one.

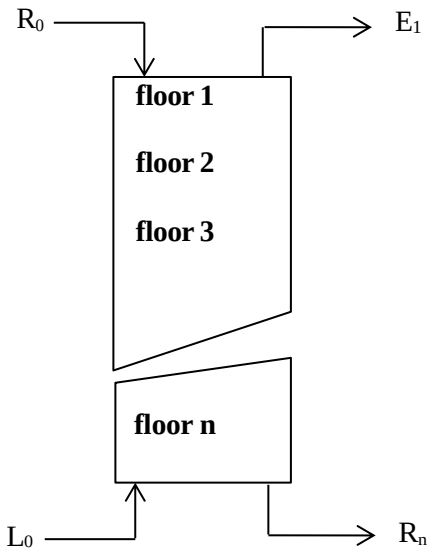

Fig. II.14. Schematic of a countercurrent multi-contact extraction system.

In this type of operation, the dense phase must be fed into the top of the column and the less dense phase into the bottom of the column.

A. Mass balance of the system

$$R_0 + L_0 = E_1 + R_n$$

$$R_0\, x_0 + L_0\, y_0 = E_1\, y_1 + R_n\, x_n$$

B. Material balance by floor

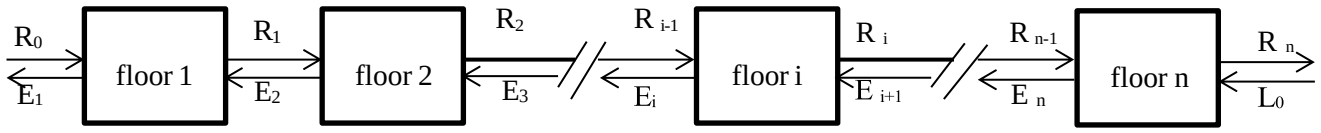

Fig II.15. Schematic of a countercurrent multiple-contact.

- **Overall material balance:**

$R_0 + E_2 = R_1 + E_1$ $R_0 - E_1 = R_1 - E_2$

$R_1 + E_3 = R_2 + E_2$ $R_1 - E_2 = R_2 - E_3$

$R_{i-1} + E_{i+1} = R_i + E_i$ $R_{i-1} - E_i = R_i + E_i$

$R_{n-1} + L_0 = R_n + E_n$ $R_{n-1} - E_n = R_n - L_0$

Hence: $R_0 - E_1 = R_1 - E_2 = \ldots = R_{i-1} - E_i = \ldots = R_n - L_0 = \Delta$ ***(the net flux through the stage, depending on the direction of stages 1 ----------> n; it is the same for all stages)***.

- **Partial material balance:**

$R_0\, x_0 - E_1\, y_1 = R_1\, x_1 - E_2\, y_2 = \ldots = R_{i-1}\, x_{i-1} - E_i\, y_i = \ldots = R_n\, x_n - L_0\, y_0 = \Delta W$ ***(the net across the floor depending on the direction of floors 1 ---> n)***.

- **Example: Net mass balance for solute A: any stage i:**

$R_{i-1}\, x_{i-1\,A} - E_i\, y_{i\,A} = \Delta W_A$ so $\mathbf{W_A = R_{i-1}\, x_{i-1\,A} - E_i\, y_{i\,A} / \Delta = constant}$

Therefore, **W_A is the net solute composition flowing through all stages**.

C. The Ponchon and Savarit method

This method is used to determine graphically the number of stages based on the material balance.

You need to know:

The mass fractions at the top and bottom of the column in each of the phases present (i.e.):

- R_0: the feed (amount of the binary mixture A + D);
- L_0: the amount of pure solvent;
- R_n: the quantity of the raffinate at the column outlet;
- E_1: the quantity of extract at the column outlet;

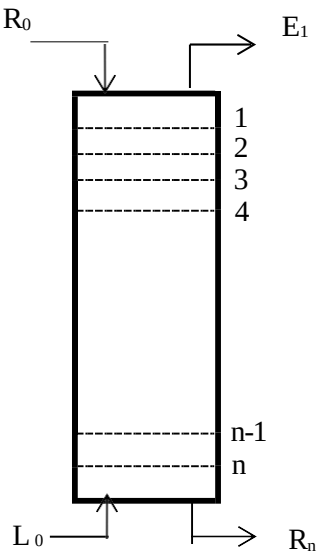

Fig II.16. The two ends of the column.

- $R_0 - E_1 = R_n - L_0 = \Delta$; therefore, by the rule of aligned points, Δ lies on the line R_0E_1 and R_nL_0.

Using the diagram in figure II.17 :

- We draw the line R_0 E_1 and extend it (**Δ must lie on this line)** – rule of aligned points;
- Draw the line R_n L_0 and extend it straight; the point of intersection is point **Δ**;
- Knowing E1, we can determine R_1 (*see II.7.1 D or II.7.2 B*);
- Next, draw the line R_1 **Δ,** which intersects the equilibrium curve at E_2;
- Knowing E_2, we determine R_2, and so on until we arrive at a value close to Rn; the number of stages is the number of segments connecting R and E.

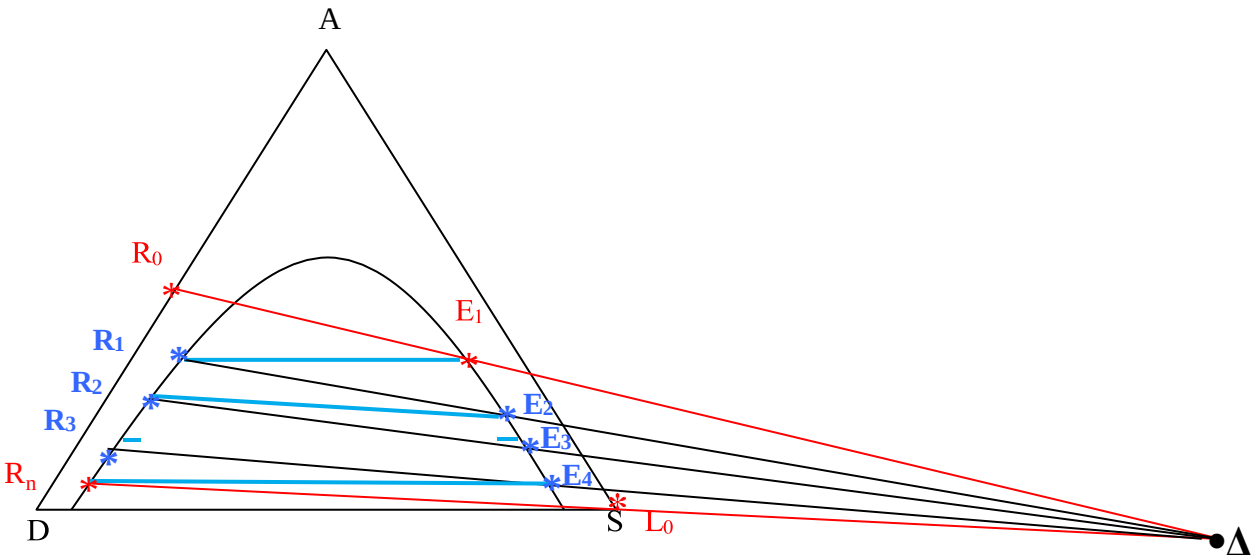

Fig II.17. Determination of the number of floors using the Ponchon and Savarit.

Note:

Since the purpose of a liquid-liquid extraction is to transfer the solute from the solution into the extract, the yield of the operation is given by the following expression: $\eta = E_1 * y_{1A} / R_0 * x_{0A}$

II. 8. Extraction in the case of total immiscibility of the solvent and diluent

II. 8. 1. Continuous co-current extraction - Formula for calculating the theoretical number of stages

Consider the following schematic diagram:

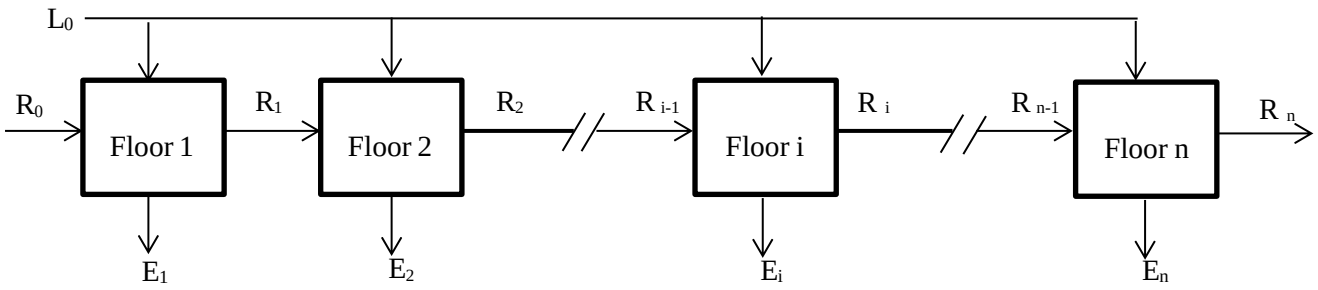

Fig II.18. Schematic of a co-current multiple-contact extraction.

The solvent (S) and the diluent (D) are immiscible with each other, so: $\mathbf{x_S = y_D = 0}$

Mass balance:

- 1^{ie} stage:

$$\begin{cases} R_0 + L_0 = R_1 + E_1 \\ R_0\, x_{0A} + \mathbf{L_0\, y_{0A}} = R_1\, x_{1A} + E_1\, y_{1A} \\ R_0\, x_{0D} + \mathbf{L_0\, y_{0D}} = R_1\, x_{1D} + \mathbf{E\, y_{1D}} \\ \mathbf{R_0\, x_{0S}} + L_0\, y_{0S} = \mathbf{R_1\, x_{1S}} + E_1\, y_{1S} \end{cases} \Rightarrow \begin{cases} R_0 + L_0 = R_1 + E_1 \\ R_0\, x_{0A} = R_1\, x_{1A} + E_1\, y_{1A} \\ R_0\, x_{0D} = R_1\, x_{1D} \quad \text{.....(9)} \\ L_0\, y_{0S} = E_1\, y_{1S} \quad \text{.....(10)} \end{cases}$$

- D present only in the load (equation 09):

$$\begin{cases} \text{Stage 1 : } R_0\, x_{0D} = R_1\, x_{1D} \\ \text{Stage 2 : } R_1\, x_{1D} = R_2\, x_{2D} \\ \cdots\cdots\cdots \\ \cdots\cdots\cdots \\ \text{Stage i : } R_{i-1}\, x_{i-1D} = R_i\, x_{iD} \\ \cdots\cdots\cdots \\ \cdots\cdots\cdots \\ \text{Stage n : } R_{n-1}\, x_{n-1D} = R_n\, x_{nD} \end{cases} \Rightarrow R_0\, x_{0D} = R_1\, x_{1D} = R_2\, x_{2D} = \ldots = R_i\, x_{iD} = \ldots = R_n\, x_{nD} = D$$

Let D be the mass of the diluent

- S present only in the effluent from each stage.

Following the same procedure as before, we find: (equation 10)

$L_0\, y_{0S} = E_1\, y_{1S} = E_2\, y_{2S} = \ldots = E_i\, y_{iS} = \ldots = E_n\, y_{nS} = L_0$; *Let L_0 be the mass of the solvent*

- For a stage (i) :

$R_{i-1} + L_{i-1} = R_i + E_i$

$R_{i-1}\, x_{i-1\,A} = R_i\, x_{iA} + E_i\, y_{iA}$ ……(11)

$R_i\, x_{iD} = D$ ⇒ $\mathbf{R_i = D / x_{iD}}$

$E_i\, y_{iS} = L_0$ ⇒ $\mathbf{E_i = L_0 / y_{iS}}$

Substituting R_{i-1}, R_i, and E_i in terms of D and L_0 into equation **(11)** for the solute, we find:

$$\mathbf{D * \underbrace{(x_{i-1\,A} / x_{i-1\,D})}_{X_{-1}} = D * \underbrace{(x_{i\,A} / x_{i\,D})}_{X_i} + L_0 * \underbrace{(y_{i\,A} / y_{i\,S})}_{Y_i}}$$

If we make a change of variables: $X_i = x_{i\,A} / x_{i\,D}$ et $Y_i = y_{i\,A} / y_{i\,S}$

$$\mathbf{Y_i = - D/L_0\ (X_i - X_{i-1})}$$

If we plot **Y** as a function of **X**, we find a line with slope m; therefore, let Y = mX, which gives: $m\, X_i = - D/L_0\ (X_i - X_{i-1})$

Substituting Xi, we find: $X_i = \frac{X_{i-1}}{m \times \frac{L_0}{D} + 1}$

Applying this relationship to the entire n-story system: $X_n = \frac{X_0}{\left[m \times \frac{L_0}{D} + 1\right]^n}$

Consequently, we determine the theoretical number of stages n: $n = \frac{Ln \frac{X_n}{X_0}}{Ln\left[m \frac{L_0}{D} + 1\right]}$

Such that: **m** is the partition coefficient (we have assumed that Y=f(X) is a line analogous to y=f(x) and consequently y = m*x, which gives m = k = y/x)

II. 8. 2. Continuous countercurrent extraction—MacCabe and Thièle construction

This method, known as the MacCabe-Thiele diagram, is used to determine the number of equilibrium stages in a system. This method is applicable only if the two liquids—the diluent and the solvent—are completely miscible with each other, a ternary diagram is not required.

A. Use of the MacCabe-Thiele diagram

The construction involves plotting two points, each representing the two ends of the apparatus on the partition curve:

- one with coordinates **(x_n, y_0)** representing the raffinate outlet and the solvent inlet;
- the other with coordinates **(x_0, y_1)** representing the feed inlet and the extract outlet;
- These two points are then connected by a straight line, called the operating line;
- Finally, stair steps are drawn between the working line and the equilibrium curve, starting at point **(x_0, y_1)** and continuing until the point **x_n** is passed. The number of steps determines the number of stories or the NET (for number of theoretical stories) of the column.

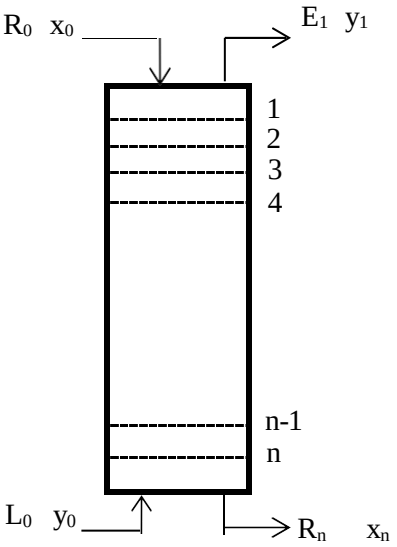

Fig II.19. The two ends of the column.

These are all of composition A. This letter is omitted to simplify the representation.

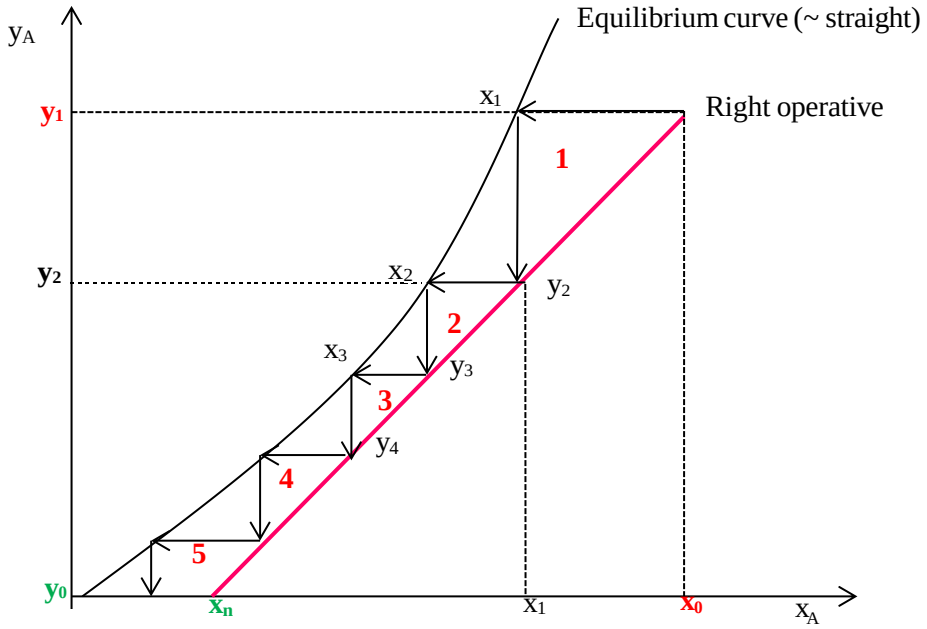

Fig. II.20. Mac Cab and Thiele diagram for calculating the number of stages in a continuous extraction system.

Note:

$E\,y_1 - R\,x_0 / E$ is negative: the transfer of A occurs from R to E

$E\,y_1 - R\,x_0 / E$ is positive: the transfer of A occurs from E to R

Note: the efficiency $\eta = \frac{m_A^E}{m_A^{Inicial}}$

Where m_A^E is the mass of solute extracted, and $m_A^{(incial)}$ is the initial mass of solute

II. 9. Extraction methods:

An extraction can be simple; in this case, it consists of thoroughly mixing three substances forming two phases at equilibrium that can be separated. This extraction corresponds to the batch extraction method.

Multiple extractions can be performed when one wishes to significantly deplete a substance of a solute or to significantly enrich a solvent with that solute. These extractions can be carried out continuously.

II. 9. 1. Discontinuous extraction apparatus:

Mixing and phase separation can be carried out in the same apparatus, known as a mixer-decanter (**see Fig. II.21**). The mixer (agitator) is selected based on viscosity, as high viscosities make mixing and separation difficult.

The decanter is a simple vessel, sometimes fitted with packing or containing a few baffles (to increase turbulence for drainage). Results obtained with industrial equipment show that a single unit (mixer-decanter) has an efficiency ranging from 0.75 to 0.9 per theoretical stage. The main drawbacks of these units are their bulk; standard installations typically comprise 4 to 8 units, though 14-unit installations do exist.

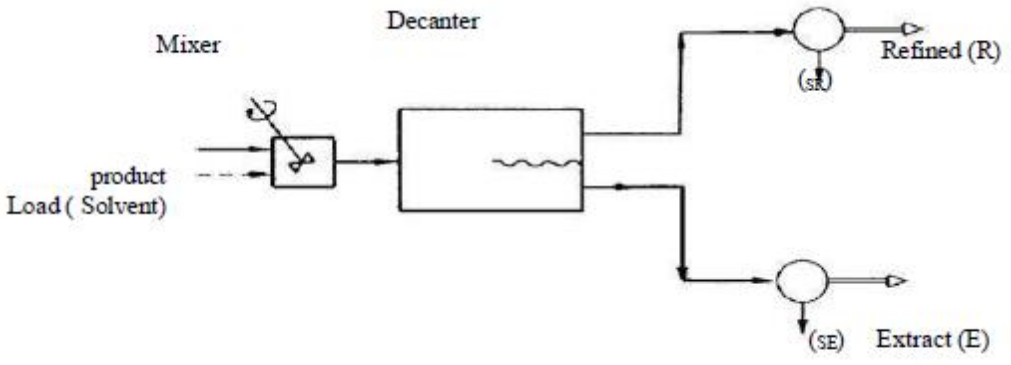

Fig. II.21. Schematic of a batch extraction setup (single-stage).

This method is also used in the laboratory with separating flasks and very rarely in industry (**see Fig. II.22**).

Despite good agitation of the liquids, with efficiency close to that of a theoretical stage, separation is relatively limited or requires large quantities of solvent.

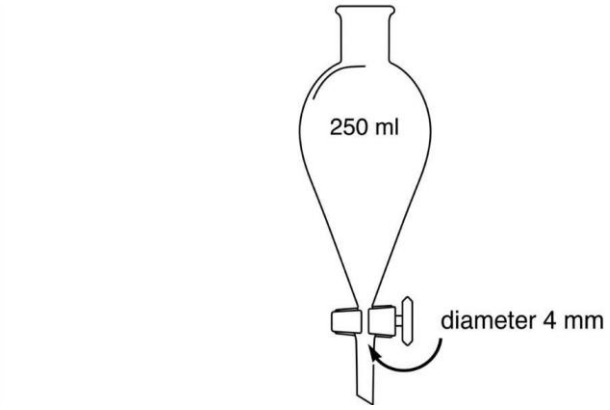

Fig. II.22. Schematic of a decanter.

II. 9. 2. Continuous extraction apparatus

Continuous extraction is performed in columns or a series of mixer-decanters; the following figures show some examples.

A. Series of mixer-decanters: In the case of multiple successive extractions, a series of mixer-decanters operate, for example, in countercurrent mode, where the extract exits at the feed inlet and the raffinate exits at the solvent inlet (**see Fig. II.23**).

In this case, the raffinate exiting the unit after decanting is directed into a second unit where it comes into contact with fresh solvent for a new operation, and so on.

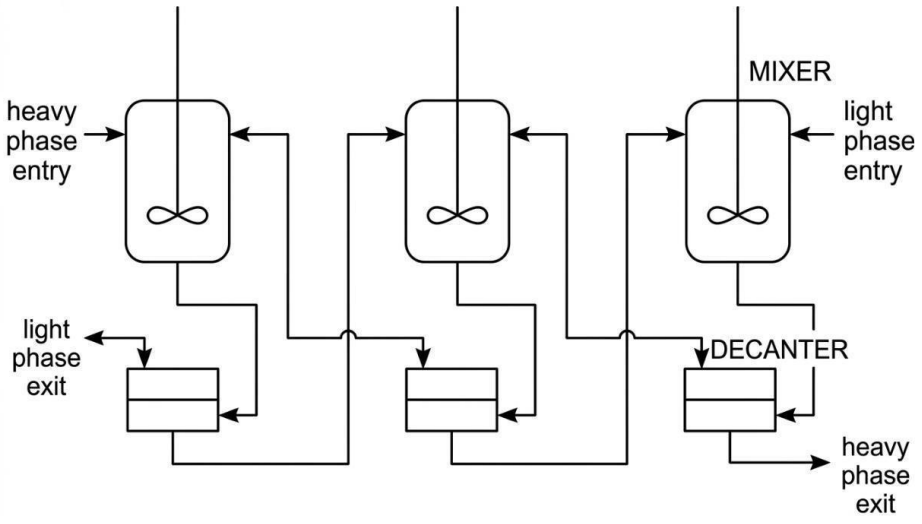

Fig. II.23. Group of counter-current mixer-decanters.

- **Figure II.24** shows a Krebs-type mixer-decanter train characterized by the presence of a chute to partially separate the phases before they enter the decanter.

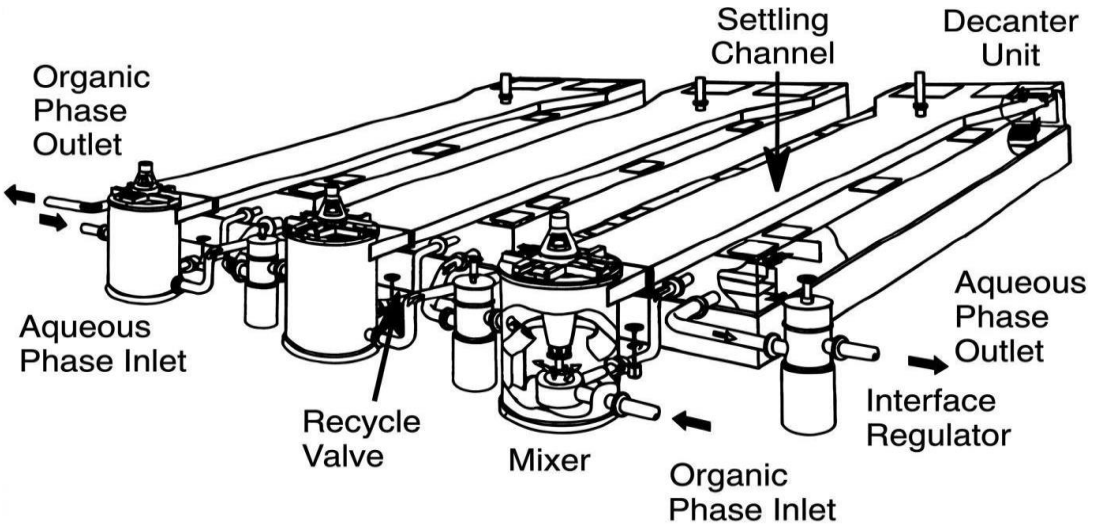

Fig. II.24. Large-scale 'Krebype' mixer-decanter unit.

B- RDC-type column: (Rotating Disc Contactor: RDC). Agitation is provided by a central rotor (agitation shaft) on which discs are mounted (**see Fig. II.25 and Fig. II.26**).

Figure II.26 shows two distinct zones: the agitation zone, also known as the transport zone, contains rotating discs to mix the phases, and the calm zone, known as the mixing zone, contains fixed rings for settling.

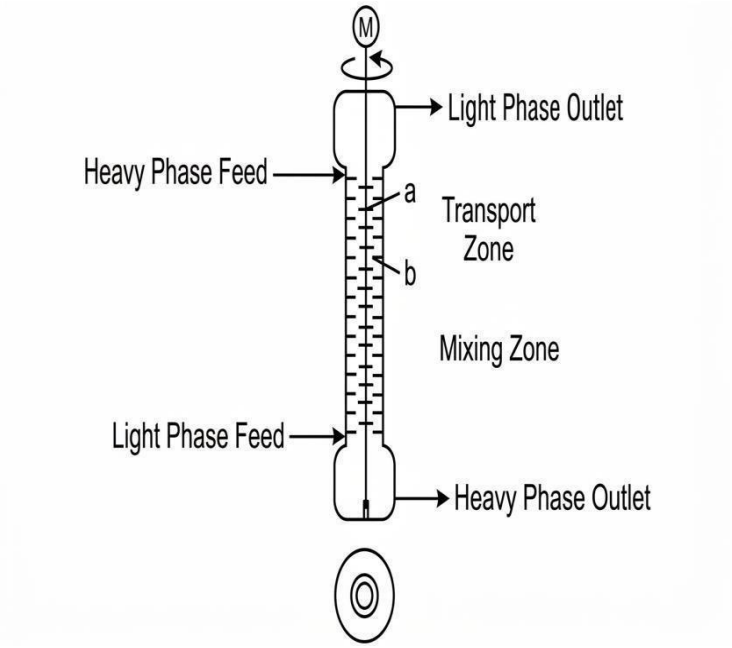

Fig. II.25. Typical RDC column.

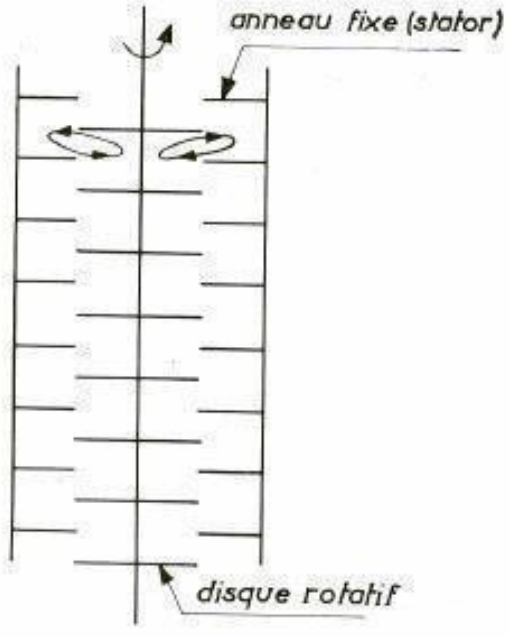

Fig. II.26. Rotary disc contactor (RDC).

C- Figure II.27 shows a spray column, a simple tube with liquid inlet and outlet.

Into which the heavy liquid is introduced at the top in the form of small droplets. These droplets descend toward the bottom of the column, passing through the layer of light liquid that flows continuously.

It is possible to operate a column of this type in reverse, that is, by spraying the light liquid at the bottom of the column, whose droplets will then pass through the continuous layer of heavy liquid rising from the bottom of the apparatus.

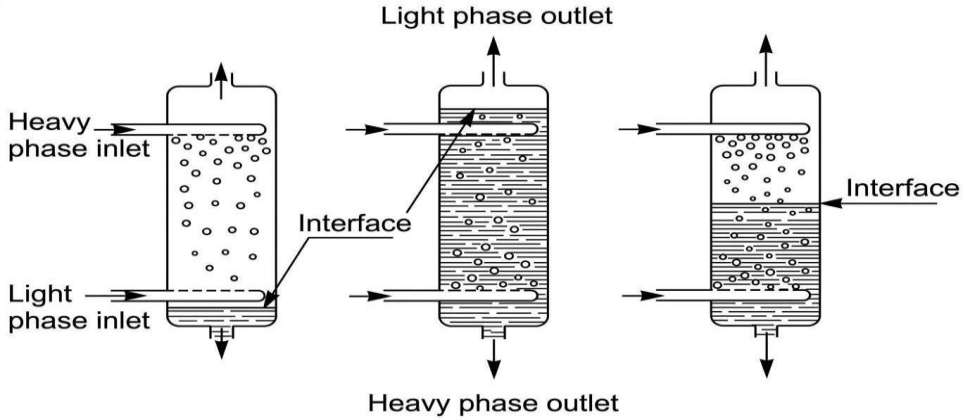

Fig. II.27. Spray columns.

D- Baffle towers: These are vertical columns packed with baffles arranged to increase liquid turbulence. These baffles can have different shapes and positions, as shown in **Fig. II.28. a, b, c**. The spacing between baffles typically ranges from 10 to 15 cm.

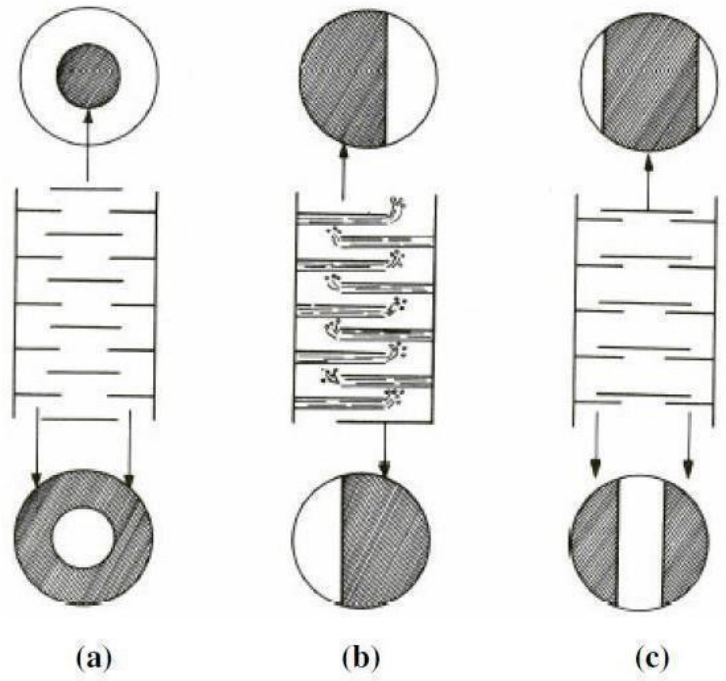

Fig. II.28. Baffle towers.

E- Pulsating column: Instead of agitation with a central axis, agitation can also be achieved by sending regular pulses. The mechanical pulsation device (bellows or pump) can be attached to a packed column to increase the turbulence of the liquids within the column (**see Fig. II.29**).

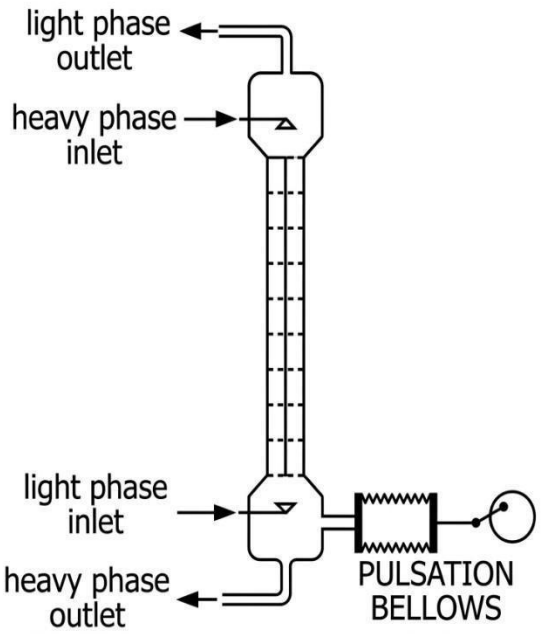

Fig. II.29. Pulsating column.

F- Perforated-tray column: Tray columns consist of trays spaced 5 cm apart and perforated with holes 2 to 3 mm in diameter, with the perforated area accounting for approximately 20% of the surface (**see Fig. II.30**).

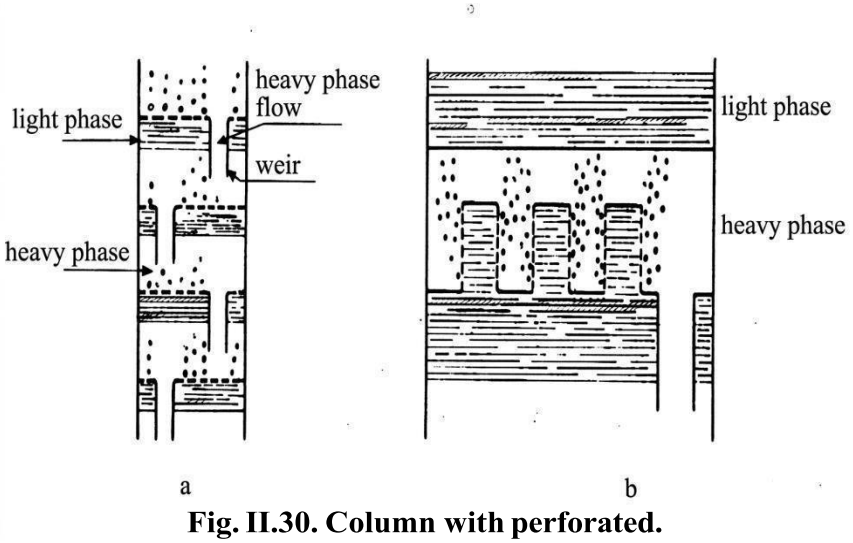

Fig. II.30. Column with perforated.

Packing: *ring-shaped or structured (3D); this type of packing is typically made of metal, although plastic rings are sometimes used to reduce weight, or ceramic rings in cases involving corrosive gases or liquids. This increases the contact surface area between the liquid and gas phases.*

II. 10. Application exercises

Exercise 1

Consider the following single-stage extraction process.

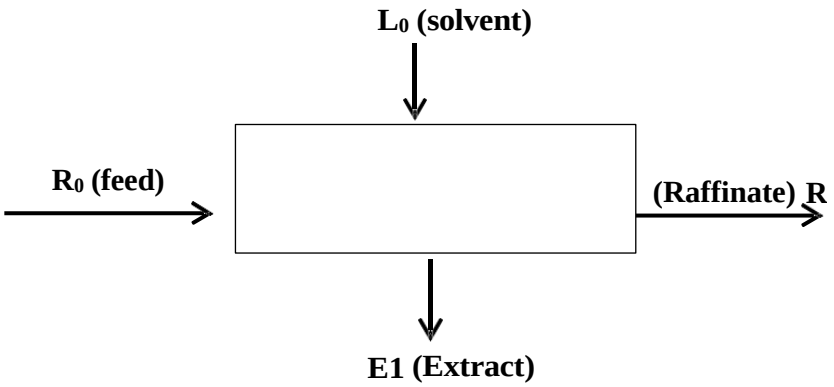

Fig. II. 31. Single-stage extraction process.

At 20 °C, 50 kg of a 20% (wt %) acetic acid aqueous solution is subjected to a single extraction stage using 90 kg of pure isopropyl ether as the extracting solvent.

1) Determine the mixture point;
2) Give the mass fractions of the streams after extraction;
3) Calculate the mass quantities of the extract and the raffinate;
4) Calculate the mass of acid extracted.

Exercise 2

Repeat the extraction described above in two stages. The amount of solvent used in each stage is 45 kg, and the same amount of solution is retained at each stage. For each stage:

1. Determine the mass fractions:
   a) Of the resulting mixture;
   b) Of the extract and the raffinate after the two phases have been separated;

2. Calculate:
   a) The mass quantities of the different streams after each extraction;
   b) The mass of acid extracted.

3. Determine the total mass of acid extracted.

Attached is the triangular diagram of the ternary mixture.

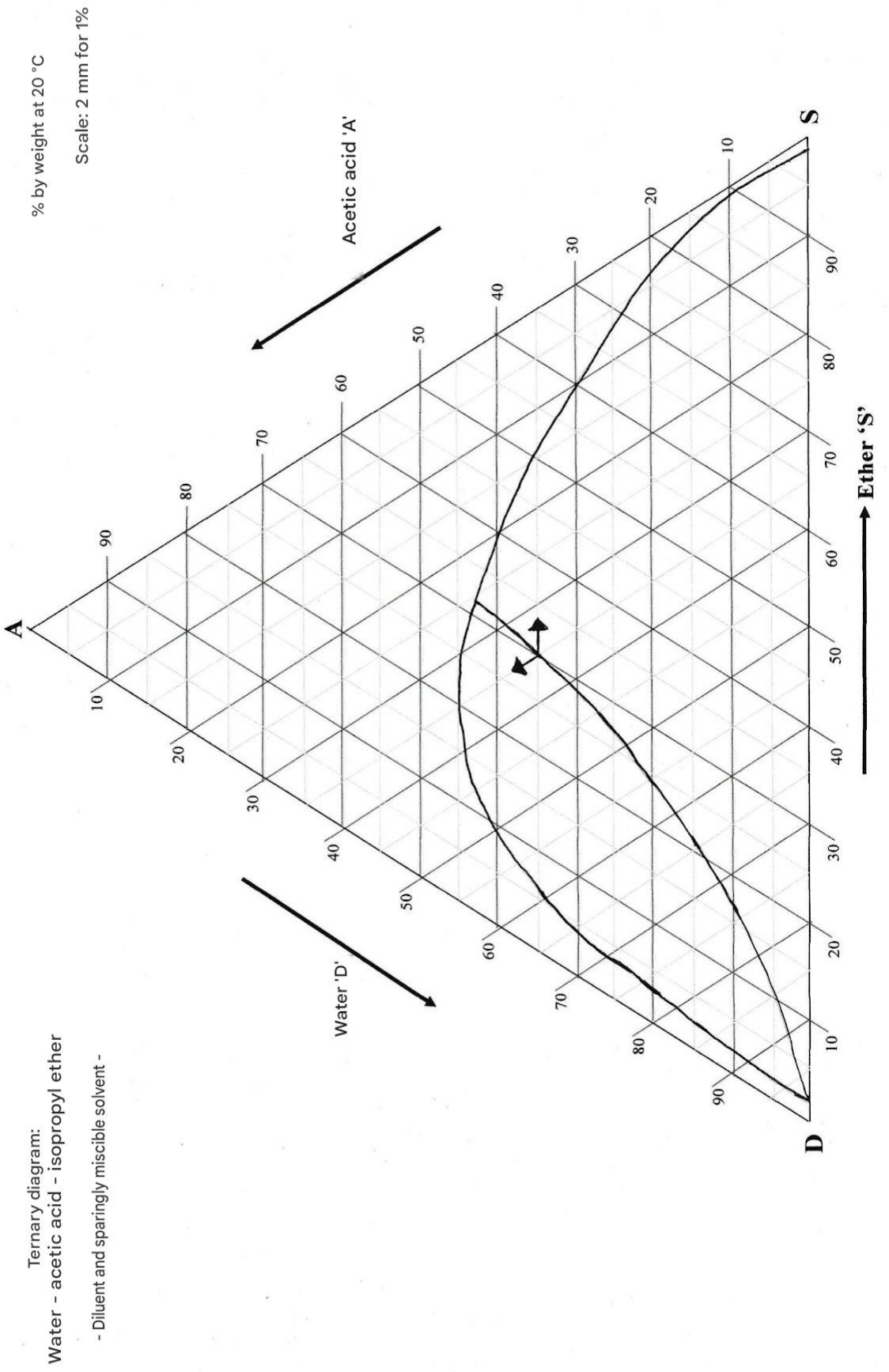

Fig. II. 32. Triangular diagram of the ternary mixture 'water, acetic acid and ether'.

Exercise 3

We wish to extract butyl acetate from an aqueous acetic acid solution using a continuous, multi-stage countercurrent extractor. The mass flow rate of the **20%** acetic acid solution feed is **100 kg/h**, while that of the solvent, which enters in countercurrent, is **200 kg/h**. The acetic acid concentration in the raffinate is **3%** and in the extract is **9%** (mass concentration).

1. Draw a diagram illustrating this extraction, given that butyl acetate is less dense than the aqueous acetic acid solution;
2. Determine the mass flow rates of the extract and the raffinate leaving the extractor;
3. Determine the amounts of acetic acid in both phases;
4. Calculate the extraction yield.

Exercise 4:

We propose to extract acetone from an aqueous solution using toluene.
The operation is carried out in countercurrent extraction column operating as follows:

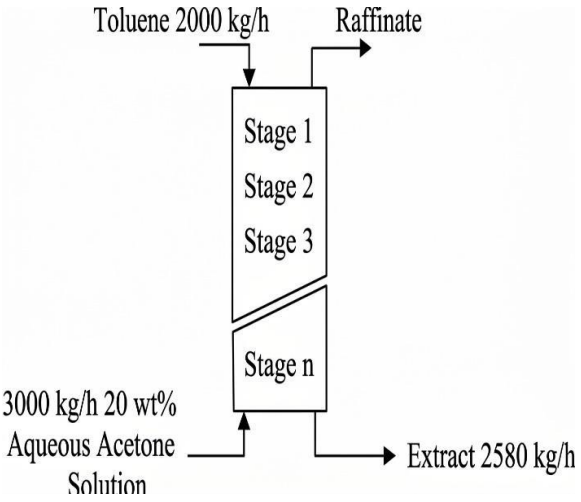

Fig. II. 33. extraction column.

1. Write the overall material balance;
2. Establish the material balance for acetone;
3. Determine the flow rate of the raffinate;
4. Determine the acetone content in the raffinate;
5. Determine the acetone content in the extract;
6. Determine the yield of the operation.

Note: water and toluene are immiscible.

Exercise 5:

A countercurrent multistage extraction system operating at 25 °C is used to recover pyridine from an aqueous solution containing **0.18** mass fraction of pyridine by extraction using chlorobenzene. The final raffinate leaving the extractor contains **0.06** mass fraction of pyridine.

- Using a graphical method, determine the number of theoretical stages required for this extraction, given that the two liquids are immiscible.

Data: Feed rate: 100 kg/h; Fresh solvent: 70 kg/h.

The equilibrium data at 25 °C for the mass fraction of pyridine in water and chlorobenzene are summarized in the following table:

Table II. 1. Mass fraction of pyridine in water.

x	y
0.000	0.000
0.035	0.020
0.077	0.050
0.097	0.070
0.120	0.100
0.135	0.125
0.145	0.150
0.152	0.175
0.160	0.200

Exercise 6

The mass compositions of the extraction of lactic acid dissolved in water by MIBK ‘methyl isobutyl ketone’ are given in the following table.

Table. II. 2. Mass compositions of lactic acid, water and MIBK in raffinate and extract.

Raffinate (%)				Extract (%)		
A	**D**	**S**		**A**	**D**	**S**
6.4	91.7	1.9		1.9	1.0	97.1
13.3	84.4	2.3		4.8	1.9	93.3
25.5	71.1	3.4		11.4	3.9	84.7
36.7	58.9	4.4		21.6	6.9	71.5
44.3	45.1	10.6		31.1	10.9	58.1
46.4	37.1	16.5		36.2	15.1	48.7

1. Questions: Plot the equilateral triangular diagram of this ternary mixture (water-lactic acid-MIBK) using the table. II. 2;

2. Application Example: We wish to extract a 35% (w/m) lactic acid solution in water with MIBK. A- Graphically determine the point M of the mixture if the quantity of solution to be treated is 1000 kg and we wish to use 420 kg of pure solvent; B- Determine the extract and the raffinate.

Chapter III.
Solid-Liquid Extraction

1. Definition;

2. Types;

3. Implementation;

4. Different Methods;

5. Example of solid-liquid extraction;

6. Extraction mechanism;

7. Solid-liquid equilibrium;

8. Janecke diagram;

9. Apparatus;

10. Application exercises.

III.1. Definition

Solid-liquid extraction is a process involving the transfer or exchange of material between a solid phase containing the substance to be extracted and a liquid phase named a solvent. Along with liquid-liquid extraction, this process is one of the liquid extraction methods.

The goal is to extract a substance present in a solid and transfer it into a liquid solvent.

The liquid used in an extraction process is called the solvent. It dissolves one or more components of the original material, known as solutes, resulting in the formation of an extract (a solution composed of the solvent and the dissolved solutes). After extraction, the solid material that remains, depleted of all or part of its extractable solutes, is referred to as the extraction residue. This residue generally consists of inert substances or compounds that are insoluble in the solvent used.

The terms "overflow" and "underflow" are used to refer to the outgoing phases from continuous-flow industrial equipment.

The extracted solution flows out from the top, and the inert solid (stationary phase) flows out from the bottom.

The transfer phenomena and the different phases resulting from this operation are shown in Figure III.1; such that:

1) Solvent (extraction liquid);
2) Stationary phase with solute —inert plus solute— ;
3) Solute;
4) Washed stationary phase —solid depleted of solute—"refining, residue, or underflow"
5) Extract—solvent with dissolved solute —“overflow.”

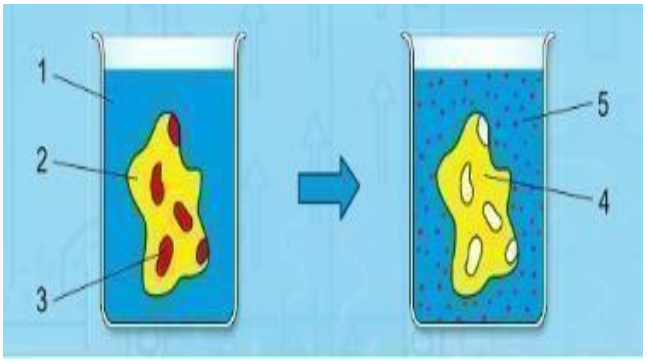

Fig. III. 1. Different phases in solid-liquid extraction.

III.2. Types:

Several solid-liquid extraction methods exist are summarised in table III. 1.

Table. III. 1. The different types of solid-liquid extraction.

Method	***Temperature of the liquid Extraction***	***Description***	***Examples of application***	***Examples of coffee preparation***
Decoction	Boiling	The solid is brought to a boil in a liquid	Tea preparation	Turkish coffee
Infusion	Close to boiling	The solid is suspended in a liquid or the solid is permeated by this liquid	How to Make Tea	French press
Digestion	Hot	The solid is placed in a liquid	Industry Pharmaceutical and perfumery	
Percolation	Usually hot	A finely divided solid is passed through a liquid		Italian coffee maker and high-pressure espresso
Leaching	Hot or cold	The finely divided solid is slowly passed through by a liquid	Hydrometallurgy and perfumery	Filter coffee
Maceration	Cold	The solid is placed in a liquid	Pharmaceutical industry and perfumery	

Elution is a form of solid–liquid extraction in which a solute adsorbed on the surface of a solid is removed by contact with a suitable liquid solvent. This process is widely used in chromatographic separations to recover or isolate compounds of interest.

Leaching is another solid–liquid extraction technique commonly employed in hydrometallurgy and environmental applications. In this process, a crushed or porous solid is contacted with a flowing solvent, either hot or cold, which dissolves and carries away the desired soluble components.

III.3. Solid-liquid extraction implementation

Solid-liquid extraction can be performed using an extractor: fixed-bed such as:

- Soxhlet extractor;
- moving-bed: moving basket extractors (Bollman) or moving-compartment extractors.

III. 4. Batch and continuous extraction methods

III. 4. 1. Batch extraction

Batch and continuous extraction are two commonly used techniques for recovering compounds from solid materials using suitable solvents under different operating conditions.

This involves maceration, which consists of soaking the solid in a solvent at room temperature, under heat, or at boiling point to extract the solid constituents. After filtration, the process can be repeated on the residue with a new portion of solvent. This method is fast but not always very efficient.

III. 4. 2. Continuous extraction

Continuous extraction takes much longer process than batch extraction, but is more efficient. Percolation: This involves slowly passing a solvent through a layer of finely pulverized substance, usually contained in a cartridge of thick, porous paper or a filter paper pouch. To ensure that the contact time between the solvent and the sample is sufficiently long, a Soxhlet extractor is used.

III. 5. Example of solid-liquid extraction

There are several examples of solid–liquid extraction, such as:

- An everyday example is the preparation of coffee.

The aromatic substances in coffee (solute) are extracted by solubilization with water (solvent) from the ground coffee.

Ideally, we obtain drinkable coffee (solvent with dissolved aromatic substances) and the completely washed coffee powder remains in the filter (stationary phase).

In reality, the stationary phase will always contain some of the solute in the solid material after extraction. Furthermore, some of the solvent will always remain bound to the stationary phase by adsorption.

The food, pharmaceutical, and fragrance industries frequently use this type of operation with a solvent: water, alcohol, or chlorinated organic solvents.

- Extraction of natural fragrances from plants: Natural fragrances are extracted from plants (flowers, roots, stems) using alcohols to produce tinctures or ointments.
- Extraction of edible oils from oilseeds: Edible oils (rapeseed, soybean, sunflower, etc.) are extracted from oilseeds by pressing followed by extraction with hexane.
- Extraction of gold and silver from ores: Gold and silver are isolated from ores by dissolution in a potassium cyanide solution.
- Copper extraction from Low-Grade Ores: Copper is extracted from low-grade ores (<1.5%) in the form of sulfate using sulfuric acid or a ferric sulfate solution.

III. 6. Solid-liquid extraction mechanism

III. 6. 1. Basic processes:

Solid-liquid extraction is carried out by bringing the solvent into close contact with the solid, followed by decanting and filtration.

It is defined as the result of the transfer of the solute present in the solid to the liquid. This transfer involves three basic processes as illustrated in Figure (III.2).

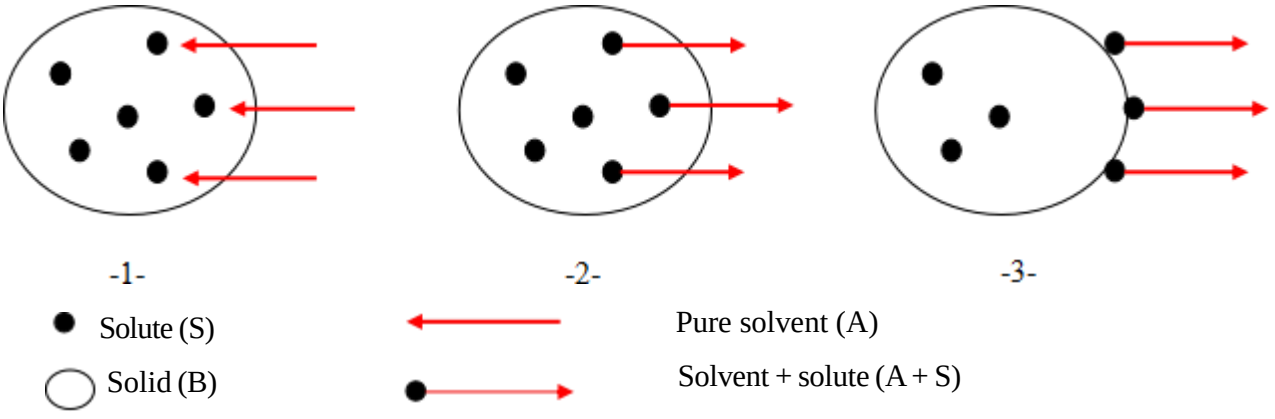

Fig. III.2. Mechanism of solute transfer during solid-liquid extraction.

1) Step 1: Passage of the solvent through the solid and dissolution of the solute within the solid particles;

2) Step 2: Diffusion of the solute from the core to the particle surface;

3) Step 3: The diffusion of the solute from the particle surface into the solvent.

III. 6. 2. Parameters affecting mass transfer

Parameters affecting mass transfer refer to the various physical, chemical, and operational factors that influence the rate and efficiency of solute transfer from one phase to another. These parameters play a crucial role in determining the performance of extraction processes. The main parameters can be summarized as follows:

1. The nature of the solvent: it must be selective, have a high dissolution capacity, a high boiling point, low viscosity, and be non-toxic, non-flammable, and non-explosive;

2. Temperature: an increase in temperature generally increases the solubility of the solute in the solvent and the diffusion coefficient of the solute within the solvent. A higher temperature may degrade heat-sensitive molecules;

3. Degree of agitation: keeping the solid in suspension homogenizes the liquid and promotes heat and mass transfer at the solid's surface.

III. 6. 3. Concept of transfer stage: mixing tank – decanter

The entire process can be broken down experimentally using a mixing-settling tank, which facilitates contact between the liquid solvent and the solid and then mechanically separates the two phases (see figure III. 3) the diagram in this figure illustrates what takes place in a batch reactor.

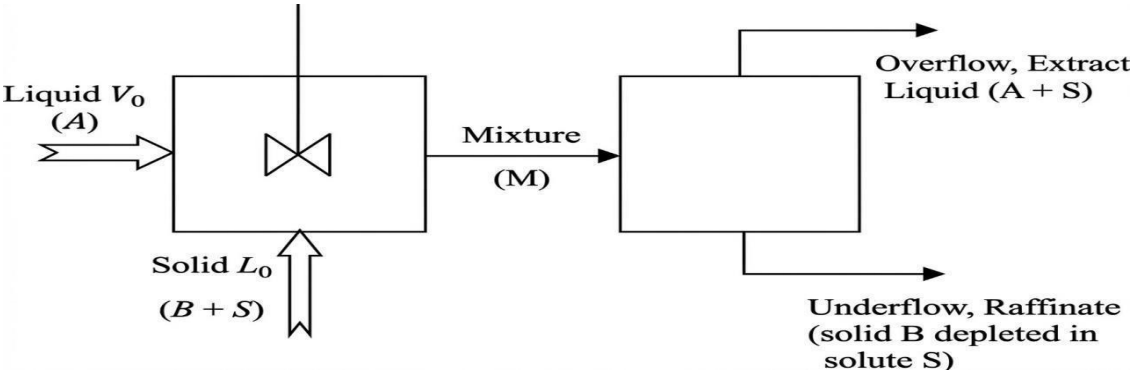

Fig. III.3. Mixing-decanting tank.

The efficiency of a mixing–decanting tank is equivalent to that of an ideal (theoretical) stage. The ideal stage is schematically represented in Figure III.4.

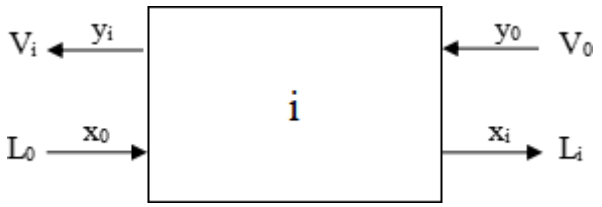

Fig. III.4. Schematic representation of a theoretical (ideal) stage.

The main terms and notations used in the transfer stage concept are as follows:

4. Concentrations y for the liquid -A: solvent -B: solid -S: solute
5. Concentration x for the solid;

L : mass flow rate of the underflow V mass flow rate of the overflow;

x_s : mass fraction of solute in the underflow;

y_s : mass fraction of solute in the overflow;

x_A : mass fraction of solvent in the underflow;

y_A : mass fraction of solvent in the overflow

X : mass ratio of solute to solution (= solute + solvent)

Y : mass ratio of inert to solution (= solute + solvent)

n : stage number or number of theoretical stages, and (i) any stage.

Mass balance

The mass balance is derived from the principle of conservation of mass. At steady state, the mass entering a system equals the mass leaving it:

Overall balance (BMG): $V_0 + L_0 = V_i + L_i$

Solute mass balance (BM/S)

$$V_0\, y_{0s} + L_0\, x_{0s} = V_i\, y_{is} + L_i\, x_{is}$$

Note: pure solvent $V_0\, y_{0s} = 0$

Balance mass on the solid (BM/B)

$$V_0\, y_{0B} + L_0\, x_{0B} = V_i\, y_{iB} + L_i\, x_{iB}$$

Note:

- *Pure solvent: $V_0\, y_{0B} = 0$*
- *Solid does not dissolve in the liquid: $Vi\, y_{iB} = 0$*

Mass balance for the solvent (BM/A)

$$V_0\, y_{0A} + L_0\, x_{0A} = V_i\, y_{iA} + L_i\, x_{iA}$$

Notes:

- *If $L_i\, x_{iA} = 0$: there is no adsorption of the solvent by the solid*
- *If $L_0\, x_{0A} = 0$: at the inlet, the solid does not contain any solvent*

III. 7. Solid-liquid equilibrium

In general, the inert solid component B may be soluble in solvent A. Using the conjugate lines of the ternary diagram on the binodal curve, it is possible to plot the distribution diagram y = f(x) characterizing the phases at equilibrium.

We then find the representations used in solid-liquid extraction, where, for a solvent A, a solid B, and a solute S, an equilibrium curve and conjugate lines characterize the equilibrium between the solid phase and the liquid phase. Since solid B is considered soluble in the solvent, the equilibrium curve does not lie on the bisector of the diagram y = f(x), and the conjugate lines are not vertical.

The main three-component system diagrams used in solid-liquid extraction are right-angled triangle diagrams.

The A'P portion of the binodal curve represents the liquid phase (y), and the B'P portion represents the solid phase (x). Starting from a mixture M, two equilibrium phases are obtained with compositions y = S / (A+B+C) and x = S / (A+B+C), representing the mass fractions of S in the liquid and S in the solid, respectively.

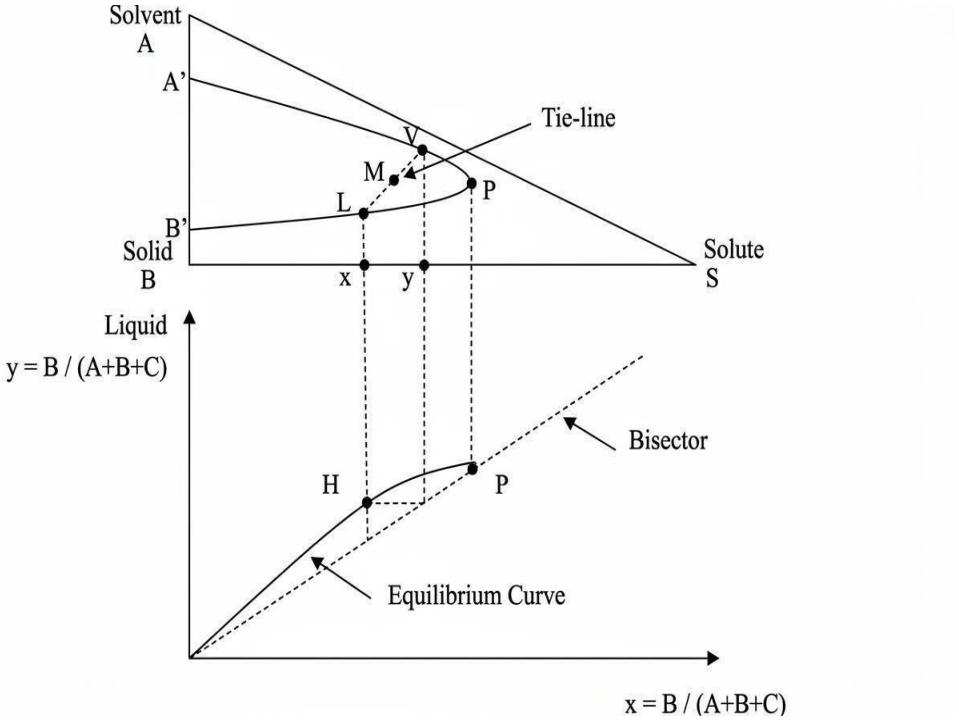

Fig. III.5. The equilibrium curve and conjugate lines.

III. 8. Janecke diagram

In the Janecke diagram (see Fig III.6), new variables are introduced to express compositions without considering the solvent. On the x-axis, we plot Xj or Yj, the compositions of the raffinate and the extract, respectively.

On the y-axis, we plot a variable Zj, which characterizes the amount of solvent.

In extraction, the solvent is added at the bottom to generate a second phase, and at the top it is separated to recover the extract and recycle the reflux, which is enriched in solute. On the Janecke diagram, the ordinate Zj, which is related to the amount of solvent, is analogous to enthalpy.

$$X_J = \left[\frac{\text{solute}}{\text{solute} + \text{diluent}}\right]_{\text{Raffinate}}$$

$$Y_J = \left[\frac{\text{solute}}{\text{solute} + \text{diluent}}\right]_{\text{Extract}}$$

$$Z_J = \left[\frac{\text{solvent}}{\text{solute} + \text{diluent}}\right]_{\text{Extract or Raffinate}}$$

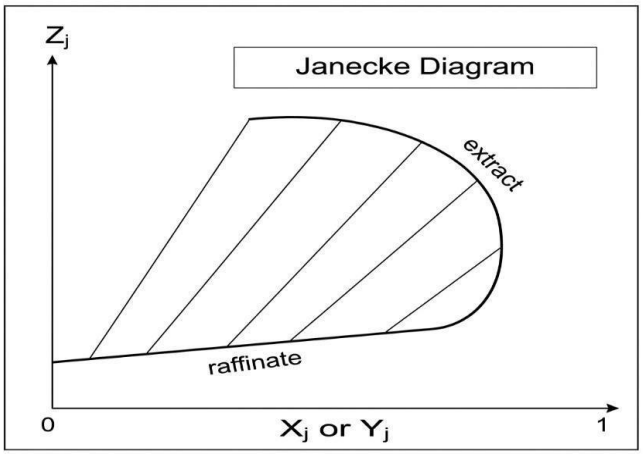

Fig. III. 6. Solid-Liquid extraction Janecke diagram.

III.9. Extraction apparatus

III. 9. 1. Batch extractors

An **open fixed-bed extractor** is a solid–liquid extraction device in which the solid material remains stationary as a fixed bed, while the solvent is continuously pumped through the bed. The solvent dissolves the desired solute from the solid particles and carries it out of the extractor. The extract solution leaves the unit through an overflow outlet, while the solid bed remains in place on a support (grate, screen, or cloth).

Main advantages: simple design, low operating cost, and easy operation. **Main limitation:** extraction efficiency may decrease due to channeling and incomplete contact between the solvent and the solid bed.

This extractor is shown in Figure III.7.

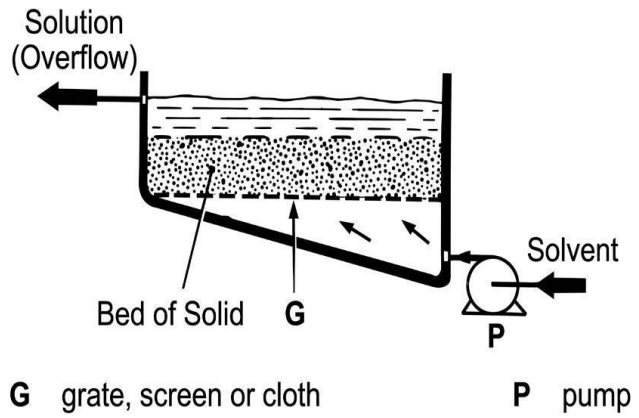

Fig. III.7. Open fixed-bed extractor.

A **Soxhlet extractor** is a laboratory apparatus used for **solid–liquid extraction** when the desired compound has limited solubility in a solvent and the solid contains insoluble impurities. The solvent is heated to reflux, evaporates, condenses above the solid sample, and repeatedly washes the solid. The extract-rich solvent is periodically siphoned back into the boiling flask, allowing efficient extraction with a relatively small amount of solvent.

Main advantages: high extraction efficiency, automatic operation, and reduced solvent consumption.

Main applications: extraction of oils, fats, natural products, pharmaceuticals, and organic compounds from solid materials.

This extractor is shown in Figure III.8.

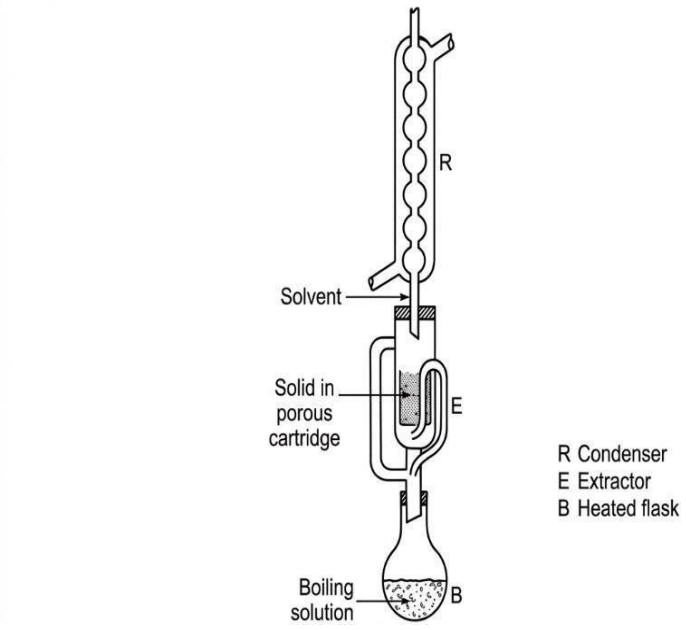

Fig. III.8. Soxhlet extractor.

III. 9. 2. Continuous extractors

A **Rotocel extractor** is a continuous solid–liquid extraction unit widely used in the food, pharmaceutical, and chemical industries. It consists of a rotating circular vessel divided into compartments (cells) containing the solid material. The solvent flows through the solids, usually in a countercurrent manner, to dissolve and recover the desired solute efficiently.

Main advantages: continuous operation, high extraction efficiency, low solvent consumption, and large processing capacity.

Main applications: extraction of vegetable oils from oilseeds (soybean, sunflower, rapeseed), recovery of valuable compounds from solids, and various chemical processing operations.

This extractor is shown in Figure III.9.

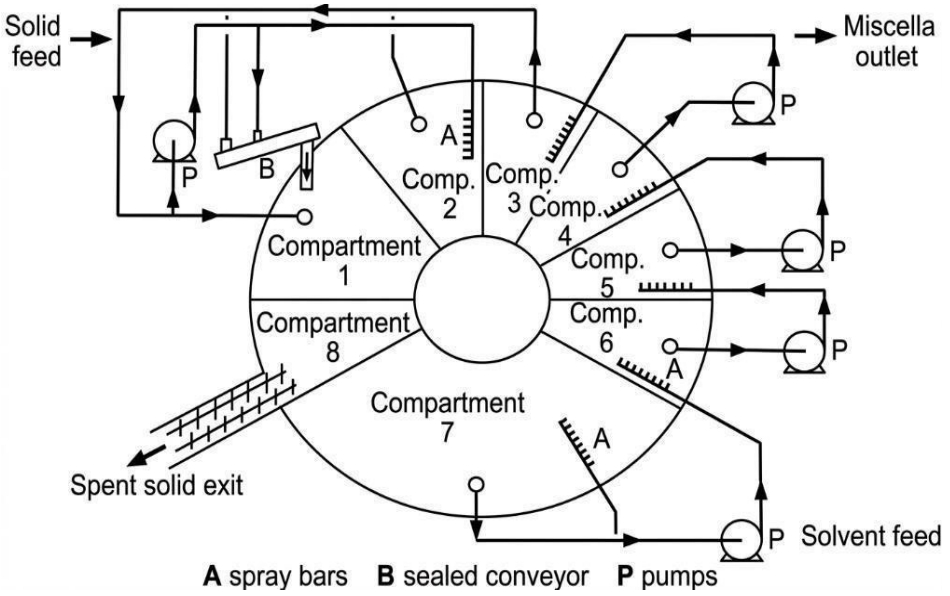

Fig. III.9. *ROTOCEL* extractor.

III. 10. Practical exercises

Exercise 1

We wish to extract, using countercurrent extraction, the soluble constituents contained in 1000 kg of sugar beets using pure hot water at a rate of 1200 kg of hot water per ton of sugar beets. Sugar beets consists of 8.5% water-soluble matter, 2.0% insoluble matter, and 89.5% water. The extraction yields 1300 kg of sugar juice containing 6% soluble dry matter.

Neglecting the phenomenon of adsorption process; determine:

1- The mass and mass composition of the pulp (solid residue);

2- The mass of the various components (solid residue);

3- The yield of the process (All concentrations are expressed by mass.)

Exercise 2

100 tonnes of ore containing 15% solute and 5% moisture (by mass) are treated with 100 t of pure water in a multistage countercurrent system comprising three ideal stages. The underflow from each stage contains 0.3 kg of solution (solvent and solute) per kilogram of inert material. Using the given data, determine the following:

1- The mass of underflow recovered at each stage;

2- The mass of overflow leaving each stage;

3- The percentage of solute recovered;

4- The composition of the overhead.

Chapter IV.
Absorption and Stripping

1. Principle of desorption;

2. Types of absorption;

3. Conditions for absorption;

4. Vocabulary;

5. Industrial implementation;

6. Application examples;

7. Study of a gas-liquid contactor;

8. Absorption isotherm;

9. Numerical applications.

IV.1. Absorption principle

The absorption process involves the separation of a gas mixture using a solvent.

The purpose of this process is to transfer the solute contained in a gas to a liquid solvent in which the solute is soluble.

This is done either to purify a gas of certain undesirable components or to recover a desired gas from the mixture.

The absorption process is facilitated by high pressure and low temperature, making it an exothermic reaction (heat is released).

IV.2. Desorption principle

The reverse process of absorption, i.e., the removal of a solute dissolved in a liquid using a gas that is insoluble in that liquid (air, water vapor, inert gas: nitrogen).

This is generally an endothermic reaction (heat absorption), and it is favored by low pressure and high temperature.

IV.3. Absorption types

IV.3.1. Physical absorption

If the compound being transferred undergoes no change, the process is simple absorption; this is physical absorption based on the compound's solubility in the liquid phase

IV.3.2. Chemical absorption

In some cases, the absorption process is accompanied by a chemical reaction in the liquid phase; this is referred to as chemical absorption (the gas reacts with the absorbing liquid to form a more or less stable substance; gas scrubbing: H_2O, SO_2, CO_2).

Note:

The solubility of gases in the solvent increases as their partial pressure increases.

The solubility of the gas is greater as the temperature of the solvent decreases; that is, the colder the solvent, the greater the solubility of the gas.

IV.4. Conditions for absorption

The successful operation of an absorption process depends on the following conditions:

IV.4.1. Temperature

Solubility increases as temperature decreases. It is therefore advisable to work at as low a temperature as possible. Furthermore, it is important to note that absorption is an exothermic process and that the heat generated must be dissipated.

IV.4.2. The contact surface:

By increasing the contact surface area between the gas and the liquid, the potential for exchange between the two phases is increased.

IV.4.3. Contact time

The contact time must be sufficient to allow the transfer of solutes from the gas phase to the liquid phase.

IV.4.4. Choice of solvent

The selection of an appropriate solvent is a key factor in absorption processes:

- ✓ It must not be viscous;
- ✓ It must not produce foam;
- ✓ Must be low in volatility to minimize entrainment by the gas;
- ✓ Must have a high absorption capacity;
- ✓ Must be easy to separate from the product to be absorbed;
- ✓ In general, the solvent must be thermally stable, inexpensive, non-toxic, and non-flammable.

IV.5. Vocabulary

Understanding adsorption requires familiarity with key terms such as:

1. **Absorber:** absorption column (gas-liquid contactor, scrubber);
2. **Desorber:** desorption column (degasser);
3. **Absorbate:** gas to be recovered from the gas phase by dissolution in the liquid through absorption (solute);
4. **Diluent:** gas that is poorly soluble or insoluble in the liquid (inert); gas containing the solute during absorption;

5. **Rich gas:** gas feed in absorption, gas outlet in desorption;
6. **Lean gas:** output from absorption, gas feed to desorption;
7. **Solvent or absorbent:** liquid feed intended to dissolve the solute in absorption;
8. **Rich solution:** liquid outlet rich in solute during absorption.

IV.6. Industrial application

IV.6.1. Equipment

Adsorption plays a crucial role in many industrial sectors due to its efficiency in separation, purification, and environmental protection processes.

Absorption requires a separation column in which mass transfer is facilitated by a large contact surface area between the liquid and the gas. Absorption columns are generally trayed or packed columns.

The choice of contactor depends on:

- ✓ Its ability to establish a perfect contact between the two phases;
- ✓ The volume of liquid being processed.

- Plate columns are of interest if:

There is a large volume of liquid (diameter > 1.5 m);

The pressure drop is not significant;

We can operate in co-current or counter-current modes;

The two phases do not form foam.

- Packed columns are of interest if:

There are many stages;

The solvent (liquid) flow rate is low;

The pressure drop is significant;

The two phases form foam.

IV. 6. 2. Diagram of an absorption column

The figure IV. 1 shows a column in which the two phases—gas mixture and liquid solvent—flow in countercurrent (or co-current) direction.

The trays or packing are designed to disperse one of the two phases within the other. Once the packing is wetted, it provides a continuous film with a very large surface area that is swept by the rising vapor.

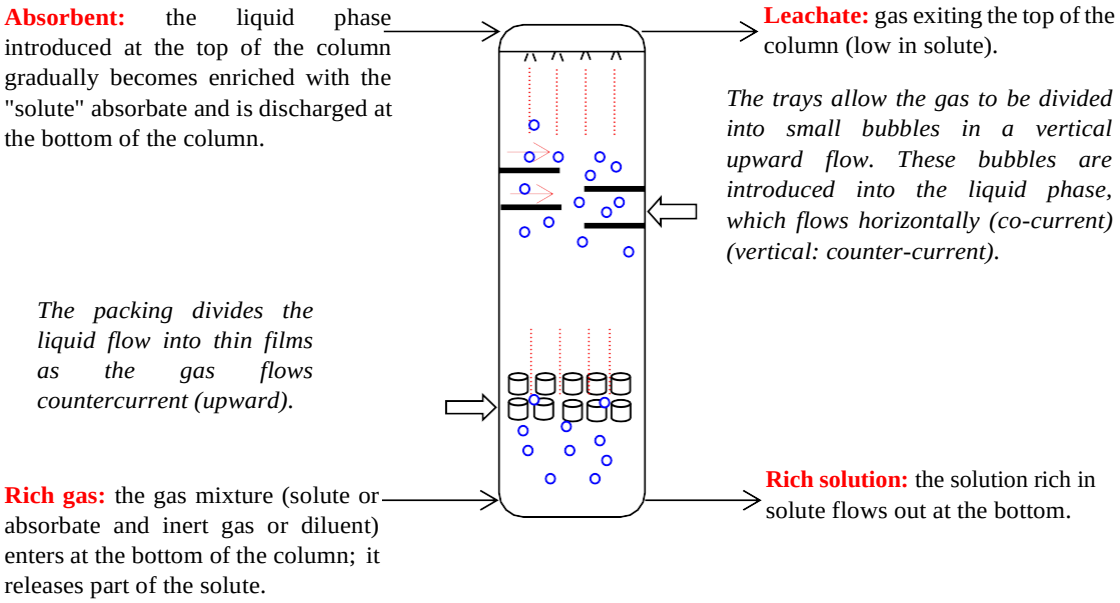

Fig. IV. 1. Absorption column.

Notations

The following symbols and abbreviations are used throughout this course to describe adsorption and absorption processes (see also Fig. IV. 2):

A. Liquid phase:

- **L:** liquid phase flow rate; index '0' at the inlet, '1' at the outlet;
- **x:** mass concentration of solute in the liquid phase;
- **(1 – x):** mass fraction of the solvent free of solute;
- **L':** flow rate of the solvent free of solute: $(1 - x) = L' / L \Rightarrow L' = L (1 - x)$;

This flow rate is constant throughout the column when the inert gas is insoluble in the liquid;

- **X:** mass fraction of the solute in the liquid phase, where $X = x / (1 - x)$.

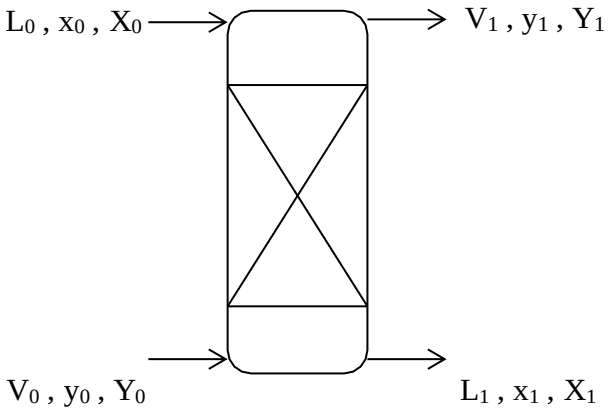

Fig. IV. 2. Mass fraction and flow rate.

B. Gas phase:

- **V:** gas phase flow rate; index '0' at the inlet, '1' at the outlet;
- **y:** mass fraction of solute in the gas phase;
- **(1 – y):** mass fraction of the inert gas excluding the solute;
- **V':** flow rate of inert gas excluding solute: $(1 - y) = V' / V \Rightarrow V' = V (1 - y)$;

This flow rate is constant throughout the column when the inert gas is insoluble in the liquid.

- **Y:** mass fraction of solute in the liquid phase, where $Y = y / (1 - y)$.

Mass balance

- **Overall mass balance**

$$L_0 + V_0 = L_1 + V_1$$

- **Mass balance / solute:**

$$L_0 x_0 + V_0 y_0 = L_1 x_1 + V_1 y_1$$

- **Mass balance / solvent:**

$$L_0 (1 - x_0) = L_1 (1 - x_1) = L'$$

- **Mass balance / inert:**

$$V_0 (1 - y_0) = V_1 (1 - y_1) = V'$$

V. 7. Absorption applications

IV. 7. 1. Example of solute

- CO_2: Carbon dioxide;
- H_2S: Hydrogen sulfide;
- NH_3: Ammonia;
- Cl_2: Dichlorine;
- HF: Hydrogen fluoride;
- SO_2: Sulfur dioxide.

IV. 7. 2. Example of a solvent

- H_2O: Water;
- Alkanolamines: These are chemical compounds containing hydroxyl (-OH) and amine (-N) groups on an alkane backbone (C_nH_{2n}):

-OH: increases water solubility (promotes water solubility);

-N: this group reacts with acidic gases (CO_2 or H_2S will bind to the –N group).

Primary alkanolamine: *a chemical compound that is both a primary amine and a primary alcohol*

- MEA: monoethanolamine (ethanolamine – 2-aminoethanol):

$\underline{(C_2H_4OH)NH_2}$ $\Longrightarrow$ **$HO\text{-}CH_2\text{-}CH_2\text{-}NH_2$** $\Longrightarrow$ **RNH_2**

(grouped under R for ease of reading)

Molecular formula (produced by reacting ethylene oxide with aqueous ammonia)

Secondary alkanolamine: *a chemical compound containing <u>a diol</u> and a secondary amine*

- DEA: diethanolamine: **$HN(CH_2CH_2OH)_2$** $\Longrightarrow$ **R_2NH**

Tertiary alkanolamine: *(or R_3N)*

- MDEA: methyldiethanolamine: **$CH_3N(CH_2CH_2OH)_2$**

IV. 7. 3 Absorption example

- The dissolution of oxygen from the air into the blood during human respiration;
- The dissolution of oxygen in water is essential for fish;
- Removal (reduction) of CO_2 ("decarbonation") contained in natural gas using an ethanolamine solvent. *Liquefied at –160 °C*

Reaction:

- **CO_2 + MEA:**

$$2\ RNH_2 + CO_2 + H_2O \rightleftharpoons \begin{matrix} RNH_3 \\ CO_3\ \text{'Carbonate'} \\ RNH_3 \end{matrix}$$

- Removal (reduction) of H_2S from natural gas ("desulfurization") by absorption; the solvent used is ethanolamine.

Reaction:

- **H_2S \MEA:**

$$2\ RNH_2 + H_2S \rightleftharpoons \begin{matrix} RNH_3 \\ S \\ RNH_3 \end{matrix}$$

- Production of precipitated calcium carbonate by reacting CO_2 with lime water:

- Calcium carbonate: $CaCO_3$: Main component of chalk and marble;
- Lime water: $Ca(OH)_2$: calcium hydroxide: A decomposition product of limestone.

Reaction:

$$Ca(OH)_2 + CO_2 \rightleftharpoons CaCO_3 + H_2O$$

- Production of sodium hypochlorite by absorption of chlorine in soda ash (bleach, NaClO).

Reaction:

$$NaOH + Cl_2 \longrightarrow NaCl + NaClO + H_2O$$

VI. 8. Study of a gas-liquid contactor

VI. 8. 1. Gas-liquid equilibrium

A system is said to be in equilibrium when three simultaneous conditions are met:

a) Thermal equilibrium: the temperature is the same at all points;

b) Mechanical equilibrium: the pressure is the same at all points;

c) Chemical equilibrium: the reaction rates of the components are the same.

The theoretical study of these equilibria is relatively complex. Nevertheless, it is possible to identify simple relationships at constant temperature.

- ***Henry's Law:***

This relationship was first stated in 1803. It can be applied to all gases as well as to liquid-gas mixtures, provided that the component is present in very small quantities (solute concentration less than 5%).

It states: "At constant temperature and for low concentrations, the partial pressure in the vapor phase of a component of a liquid-gas mixture is proportional to the molar concentration of that component in the solution."

$$\mathbf{PA = H_A X'_A}$$

- A: solute;
- P_A: partial pressure of A in the gas;
- x'_A: molar concentration of A in the liquid;
- H_A: Henry's constant.

The Henry's law constant is determined experimentally and is found in tables at various temperatures; it has the dimensions of a pressure.

- ***Dalton's law:***

The partial pressure of a component (A) in the vapor phase is equal to the product of the molar fraction in the vapor phase and the total pressure of the mixture.

$$\mathbf{P_A = P_t\ y'_A}$$

- Pt: total pressure;
- y'_A: molar fraction of solute in the gas.

$$\left.\begin{array}{l} P_A = H_A \; x'_A \\ P_A = P_t \; y'_A \end{array}\right\} \Rightarrow P_t \; y'_A = H_A \; x'_A \Rightarrow y'_A = \frac{H_A}{P_t} \; x'_A$$

And therefore: $H_A / P_t = k$: Share coefficient. Consequently, H_A / P_t is the slope of the partition curve: $y' = f(x')$.

IV.8.2. Concept of the operating line:

- To plot the operating line, you need to know the two endpoints of the column: (x'_0, y'_1) and (x'_1, y'_0) using the partition curve (the operating line lies above the equilibrium curve):

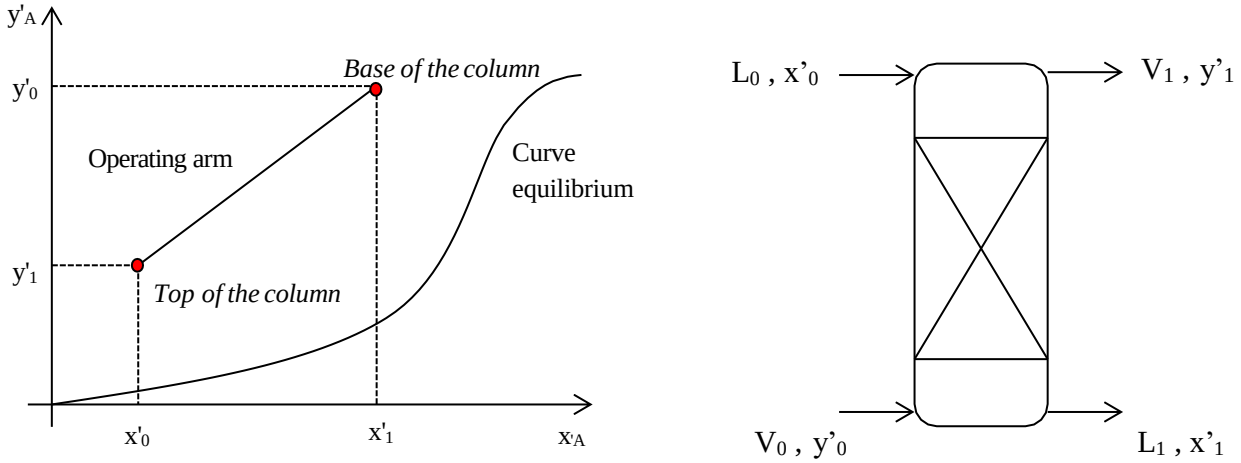

Fig. IV.3. Operating line; absorption case.

- The transfer from the liquid phase to the vapor phase is called stripping: the operating line lies below the equilibrium curve.

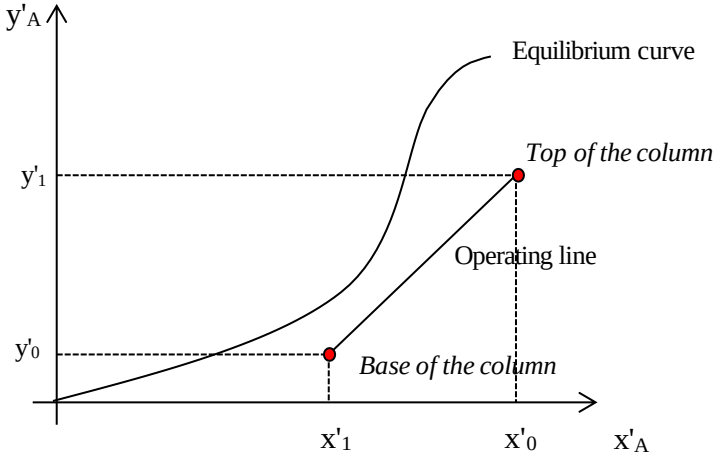

Fig. IV.4. Operational line; desorption case.

IV. 8. 3. Concept of the minimum (molar) value of Lmin / V

For the soluble component A, the mass balance equation for the entire column is expressed as:

$L_0\ x'_0 + V_0\ y'_0 = L_1\ x'_1 + V_1\ y'_1$ ..... (IV.a)

It is assumed that the molar flow rates remain constant throughout the column: (IV.a)

$\Rightarrow L\ x'_0 + V\ y'_0 = L\ x'_1 + V\ y'_1 \quad <=> \quad L\ (x'_0 - x'_1) = L\ (y'_1 - y'_0)$

$\Rightarrow \frac{L}{V} = \frac{y'_0 - y'_1}{x'_1 - x'_0} = \frac{y'_1 - y'_0}{x'_0 - x'_1}$ …….. (IV.b); slope of the operating line.

When we are in the region of very high dilution, the equilibrium curve is approximated by a straight line.

Analysis of equation (IV.b) shows that the slope of the operating line is equal to the ratio L / V; that is, the consumption of pure solvent (mole of solvent / mole of inert gas).

Assuming the initial concentration y'_0 and final concentration y'_1 of the gas are known, with a flow rate V and an initial concentration x'_0.

⇨ y'_0 ; y'_1 ; x'_0 and V are fixed.

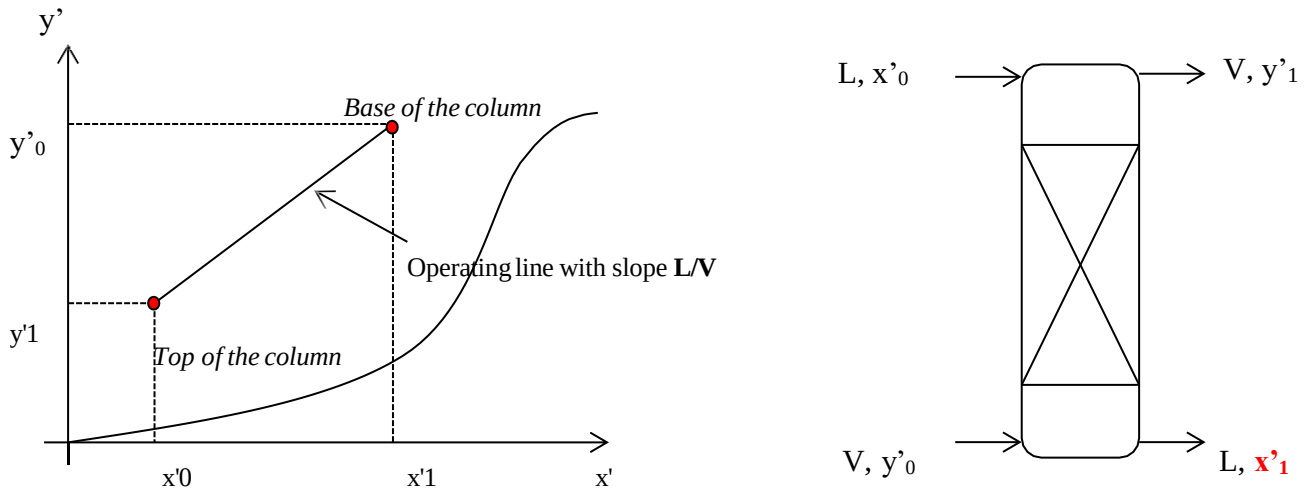

Fig. IV.5. Operating line for the L/V slope.

The value of x'_1 increases as the flow rate of solvent L decreases.

The diagram shows several positions of the operating line (for the same values of y'_0, y'_1, and x'_0) plotted as a function of different solvent consumption rates.

The ratio (L/V) cannot fall below a minimum value corresponding to the slope of the operating line that intersects the equilibrium curve; otherwise, the system enters a desorption regime.

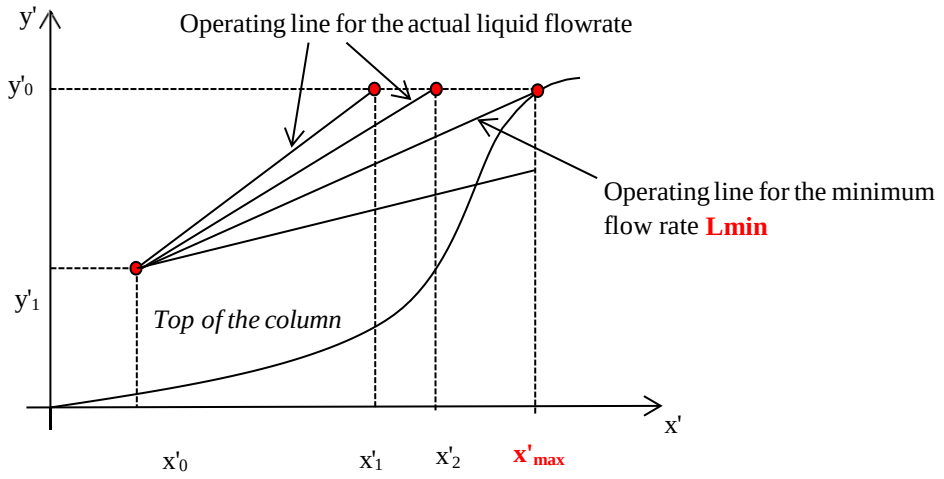

Fig. IV.6. Minimum value of the L/V ratio.

Based on economic considerations, the optimal flow rate is determined, generally lying between **1.3 L_{min}** and **1.5 L_{min}**.

Note: The minimum molar ratio of L_{min} / V is found by plotting Y' = f (X') "molar"

IV. 8. 4. Concept of number and height of theoretical plates (N.P.T.)

The calculation of an absorption column is based specifically on mass balances and phase equilibrium laws and aims to determine the number of theoretical stages required to achieve a given separation.

The number of theoretical plates required for absorption can be determined using a graphical method.

The graphical method starts with the equilibrium diagram on which the operating line is plotted (Mac Cab and Thiel method).

It consists of drawing line segments (stair steps) between the equilibrium curve and the operating line, and the number of points touching the equilibrium curve gives the minimum number of theoretical plates.

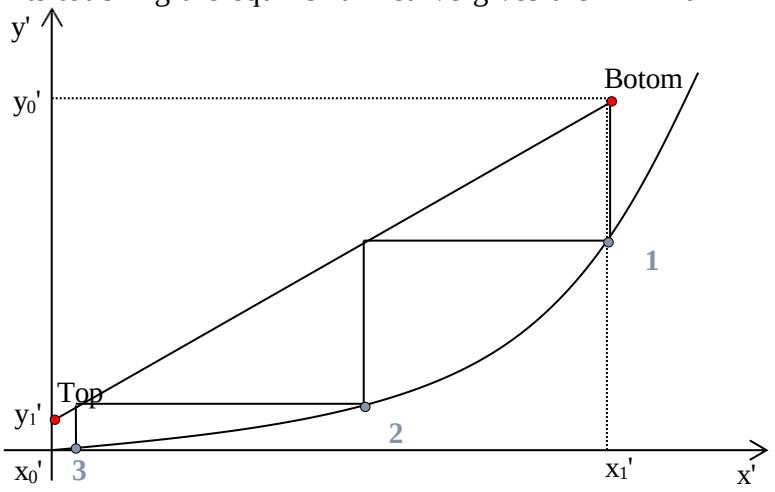

Fig. IV.7. Mac Cabe and Thiel model (pure solvent-countercurrent).

The points on the equilibrium curve represent the gas-liquid phase equilibrium on the trays, while the points on the operating line represent the state of the gas-liquid system between two positions (trays).

In practice, the number of actual trays is not identical to the number of calculated theoretical trays: ***N.P.R > N.P.T***

- E_G**:** overall efficiency of the separation column = N.P.T / N.P.R
- The height ***H*** of the column will therefore be determined by the number of actual trays N.P.R.

IV. 8. 5. Case of packed columns

Concept of HEPT:

The quality of the packing is characterized by a parameter called HEPT: the height equivalent to a theoretical plate, defined by the following equation:

H: theoretical packing height = HEPT * NPT

Correlations exist for predicting HEPT. Among these is the correlation by Harrisson and France (1989), who propose the following relationship:

$$HEPT = (1200/ap) + 4$$

Where: ap: specific surface area in ft^2/ft^3

Examples of published HEPT values (Kister 1992) can be found in tables that list packing types based on their nature and applications, along with their HEPT values in inches.

III. 9. Hydrodynamics of flows

Gas-liquid contactors are widely used in chemical, petrochemical, pharmaceutical, and para - chemical processes, as well as in wastewater and air pollution control.

The gas-liquid contactors used come in a wide variety of forms, such as tubular droplet reactors, falling film reactors, bubble reactors, tray-packed reactors, mechanically agitated tank reactors, and jet or venturi-type reactors. These different forms result from the interplay between various phenomena of chemical thermodynamics and physical and chemical kinetics that occur simultaneously and determine the choice of equipment designed to operate under optimal hydrodynamic and energy conditions. Indeed, consider the case of a gas-liquid flow

within a tubular bubble reactor of cross-sectional area Ω, where an irreversible reaction occurs between a solute AGi dissolved in the gas phase and subsequently absorbed by the liquid phase containing a reactant ALi. The reaction yields a product Pr (see Fig. IV. 8).

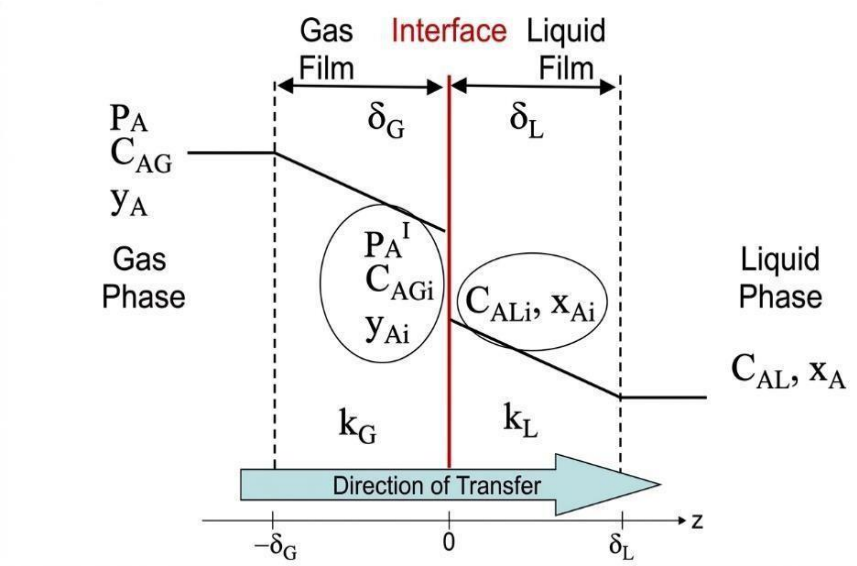

Fig. IV.8. Profile of solute concentrations in the two phases according to the double-film theory during physical absorption.

For example, oxidation with atmospheric oxygen in an organic medium. Assuming that the gas phase, with a retention rate of α, is in piston flow, and that the liquid phase, with a retention rate of β = 1 − α, is in diffusive piston flow (where D_L is the axial dispersion coefficient), a mass and heat balance in a small volume element Ωdx leads to the following.

$$-\frac{\delta G}{\Omega}\times\frac{P}{RT}\times\frac{dy_A}{dx}-Kk_G\,(Y_A-Y_A^*)=-\frac{\delta G}{\Omega}\times\frac{P}{RT}\times\frac{dy_A}{dx}-a(C_{AG}-YC_{AG}^*)=0 \quad \text{...IV.1}$$

(convection) *(transfert entre phases)*

Equations in steady state:

$$(1-a)D_L\frac{d^2C_{AG}}{dx^2}-\frac{\delta L}{\Omega}\frac{dC_{AG}}{dx}+EK_L\,a\,(C_{AG}-C_{AG}^*)+(1-a)r_A=0 \quad \text{.....IV.2}$$

(Dispersion axiale) *(conversion)* *(transfert entre phase)* *(réaction chimique)*

$$(1-a)D_L\frac{d^2C_{AL}}{dx^2}-\frac{\delta L}{\Omega}\frac{dC_{AL}}{dx}+(1-a)\,z\,r_A=0 \quad \text{.....IV.3}$$

(Dispersion axiale) *(conversion)* *(réaction chimique)*

$$D_L P_m C_{Pm} \frac{d^2T}{dx^2} - Pm\,Cm \frac{\delta G}{\Omega} \frac{dT}{dx} + (1-a) r_A \Delta H_A - \mu \vartheta (T - T_e) = 0 \quad \text{.....IV.4}$$

Equation (IV.1) means that during the convective transport of the gas, the decrease dyA in the solute concentration in the gas phase is due to the flux Φ of mass transferred to the liquid phase through the specific interfacial area a via the intermediate of the partial transfer conductances in the gas phase k_G and in the liquid phase k_L, which allow the substance AG to diffuse on either side of the interface under the influence of a transfer potential difference (C^*_{AG} - C_{AG}). In this expression of the flux, we model the "physical kinetics" of the mass transfer via the double-film model. E is the acceleration factor due to the chemical reaction, which characterizes the effect of the chemical reaction on the physical kinetics of mass transfer (E = 1 for absorption in the absence of a chemical reaction). Equation (IV.2) means that during the movement of the liquid due to axial dispersion and convection, the solute transferred from the gas phase and thus dissolved reacts with the reactant AL at a reaction rate r_A. This is the "chemical kinetics", r_A depends on the concentrations of the reactants within the liquid, for example, (within the liquid where the concentration of dissolved gas is CAG):

$$r_A = - \frac{dC_{AG}}{dx} = k_{AG\,AL}\, C_{AG}\, C_{AL} \quad \text{.....IV.5}$$

Equation (IV.3) relating to reactant B contains the same terms as the previous one, with the exception of the transfer term, assuming that reactant B is not volatile.

Equation (IV.4) describes heat transfer within a mixture of density pm and specific heat capacity C_{pm}. During the overall motion due to convection and axial dispersion, a heat flux is generated or consumed by the chemical reaction (ΔH_A is the reaction enthalpy), and a heat flux is removed or supplied from the exterior, where the temperature is T_e, via thermal conductivity (as in the case of a heating or cooling coil); ϑ is the specific heat transfer area, and μ is the overall heat transfer coefficient.

Equations (IV.1) through (IV.4) demonstrate the extent to which phenomena related to flow hydrodynamics (characterized by the retention rate of each phase and, potentially, axial dispersion within each phase).

IV. 10. Practical exercises

Exercise 1

We propose to purify an effluent containing hydrogen sulfide by passing it in countercurrent with water through a packed absorption column.

The mass flow rate of air containing hydrogen sulfide is **653 kg/h**, and the mass fraction of hydrogen sulfide is **5**%. The gas exiting the column still contains **0.12%** hydrogen sulfide (mass fraction). The mass flow rate of pure water fed into the column is **500 kg/h,** and the air is assumed to be insoluble in water.

1. Establish a total material balance;
2. Establish a material balance for hydrogen sulfide input;
3. Calculate the flow rates at the column outlet;
4. What will be the flow rate of the inert gas free of solute?
5. What will be the flow rate of the solvent without solute?
6. Determine the fraction of hydrogen sulfide recovered.
7. Calculate the absorption efficiency.

Exercise 2

Air containing **5%** acetone is fed into a packed absorption column. The mass flow rate of the air is **1000 kg/h**.

The absorbent used to capture acetone from the air is pure water. The air leaving the absorber must be free of acetone. At the absorber outlet, the water contains **4%** acetone. The bottom stream of the absorber is sent to a tray extraction column to separate the acetone from the water; this column is fed at the top with a flow rate of pure extraction solvent equal to **580 kg/h**.

The top and bottom of the extractor after extraction contain **0.5%** and **3.25%** acetone, respectively.

1. Draw and fully label a process flow diagram;
2. Calculate the flow rates of all unknown streams.

Exercise 3

When washing an air-NH_3 mixture with a molar concentration of 8% using pure water in an absorption column operating at 20 °C, the following molar concentrations of NH_3 are observed at the outlet:

- Gas phase 0.2%,
- Liquid phase 7.3%.

Determine the number of theoretical stages of the column using the attached equilibrium curve, plotted in molar ratios.

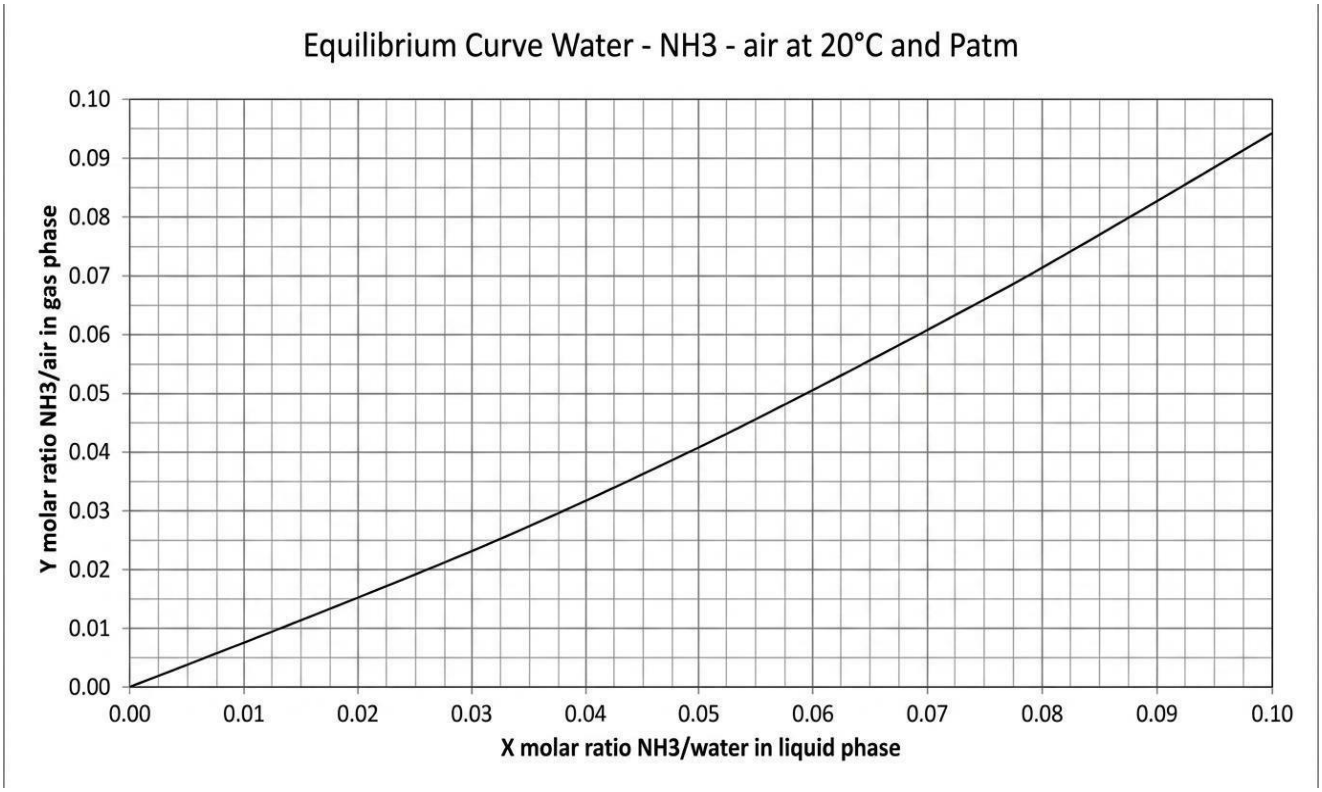

Fig. IV. 9. Equilibrium curve.

Exercise 4

We wish to recover acetone from a gas stream consisting of 14.787 Kmol/h of acetone and 9.858 Kmol/h of inert gases.

- The desired acetone absorption rate is 98%.
- This stream is treated in a tray absorption column with pure acetic acid, and the inerts are considered insoluble in acetic acid.

1) Determine the minimum molar ratio of acetic acid to inerts required to achieve this absorption;

2) Determine the molar flow rate and composition of the liquid stream (L_0 and X_1) for a solvent flow rate corresponding to 1.1 times the minimum molar flow rate.

3) Determine the number of theoretical plates and the number of actual plates, given that their efficiency is 40%.

Table. IV. 1. Solubility of acetone in acetic acid at the average temperature and column pressure, expressed in molar concentrations.

Solubility of acetone in acetic acid at the average temperature and column pressure, expressed in molar concentration													
x molar concentration of the liquid phase	0.04	0.05	0.08	0.1	0.13	0.15	0.18	0.20	0.23	0.25	0.28	0.30	0.33
Molar concentration in the gas phase	0.0015	0.005	0.010	0.012	0.043	0.075	0.180	0.270	0.367	0.420	0.495	0.536	0.590

Exercise 5

The desorption of a water-NH_3 solution using NH_3-free air is carried out continuously in a packed column. The operating conditions are as follows:

- Liquid feed rate: L_0 = 4 kg/h, mass concentration x_0 = 7%,
- Liquid flow rate: L_1 = 3.8 kg/h, x1 = 2.1%,
- Feed air flow rate: V_0 = 1.3 Nm^3/h (without NH_3).

1) Calculate the mass flow rate of desorbed NH_3 in $g \cdot h^{-11}$.

2) Calculate the mass fractions of NH_3 in the liquid phases entering and leaving the column, denoted X_0^{mass} and X_1^{mass}.

3) Calculate the molar ratios of NH_3 in the liquid phases entering and leaving the column, denoted X_0^{mol} and X_1^{mol}.

4) Calculate the molar ratio of NH_3 in the outgoing gas phase Y_1^{mol}.

Data:

- M_{air} = 29 g/mol, M_{eau} = 18 g/mol, M_{NH3} = 17 g/mol, R = 8.314 J/mol·K^1,
- Standard Conditions of Temperature and Pressure (SCTP): 0°C or 273.15 K and 1.013e5 Pa.

Chapter V.
Distillation

1. General Information;

2. Types of Distillation Operations;

3. Distillation columns;

4. Principle of distillation operation;

5. Calculation of the efficiency of a rectification column;

6. Vacuum distillation;

7. Azeotropic distillation;

8. Numerical applications.

V.1. General Information

Distillation is one of the most important separation methods. It is one of the most widely used separation processes in chemistry; this process allows for the separation of one or more components from a homogeneous liquid mixture by exploiting the difference in volatility among the components. After heating to boiling, the vapor phase rises above the boiling liquid, and through condensation of the vapor phase, a liquid called *the distillate* (also known *as the head product*) is obtained, whose composition differs from that of the initial mixture. The non-evaporated liquid phase constitutes the *residue* (also called *the tail* or *bottom product*).

The distillate is enriched with *light* components having a lower boiling point, while the residue consists of heavy components with a higher boiling point.

In fact, distillation simply consists of forming, from a liquid phase, a vapor with a composition different from that of the liquid; this vapor is immediately condensed and eventually collected as separate "fractions." The separation and the conditions necessary for its occurrence depend on the *liquid–vapor* system and, consequently, on the liquid–vapor equilibrium of the mixture to be distilled.

Among the countless applications of distillation, it is worth noting that this process forms the basis of crude oil refining.

V.2. Type of distillation process:

V.2.1. Batch distillation:

In this type of distillation, the mixture is fed into the boiler at the start of the process, and the various compounds are collected sequentially at the top of the column in order of their increasing boiling points.

- In batch distillation, a single cycle of vaporization and condensation takes place.
- It is applicable when the difference in boiling points between each component is very large, such as 25–35°C.
- It is mainly used to separate volatile liquids from non-volatile liquids.
- It is difficult to obtain pure substances through this distillation.
- The vapors produced by the boiling mixture are generally condensed.

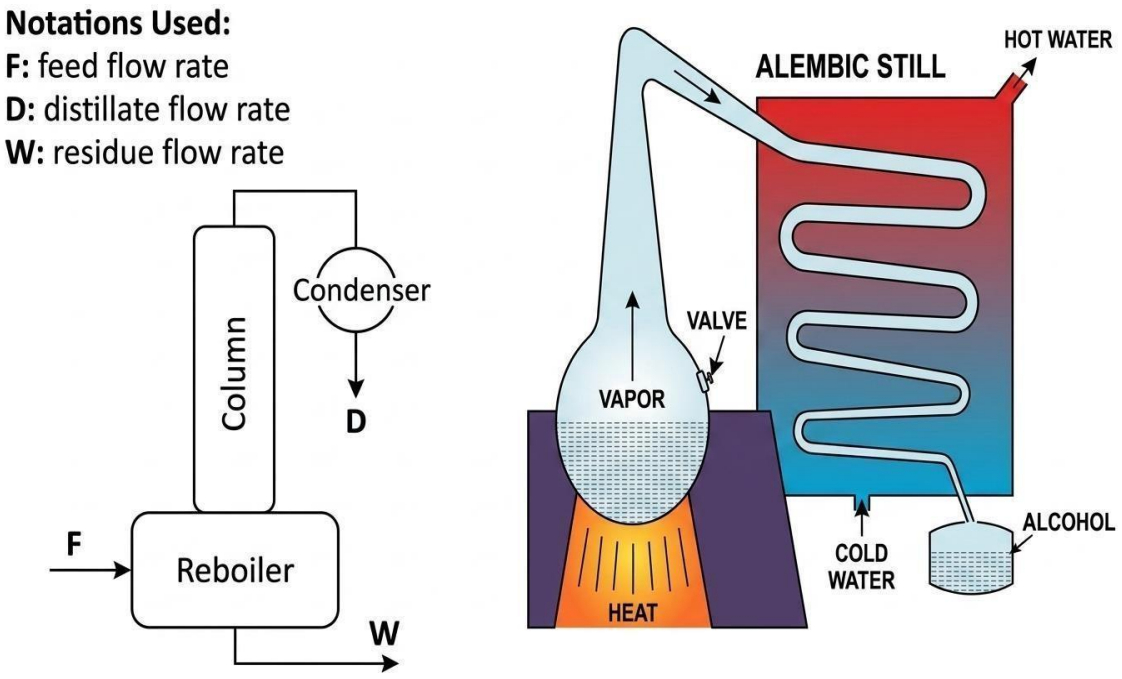

Fig. V.1. Batch distiller - column with reduced efficiency-.

V.2.2. Continuous Distillation

In continuous distillation, the feed mixture is continuously introduced into the column while the distillate and bottom products are continuously withdrawn. As a result, the process operates under steady-state conditions, and the compositions within the column remain essentially constant over time. However, in batch distillation, the composition of the liquid mixture in the still changes continuously due to the progressive removal of the more volatile component.

The continuous distillation process requires the use of a column.

V.3. Distillation Columns:

There are two main types of **continuous distillation** columns:

- **Tray columns**: these consist of a number of trays that allow the liquid, which flows down the column by gravity, to come into contact with the rising vapor.

  In co-current tray columns, the rising gas phase passes through the liquid, which moves horizontally across the tray, hence the name of this type of column.

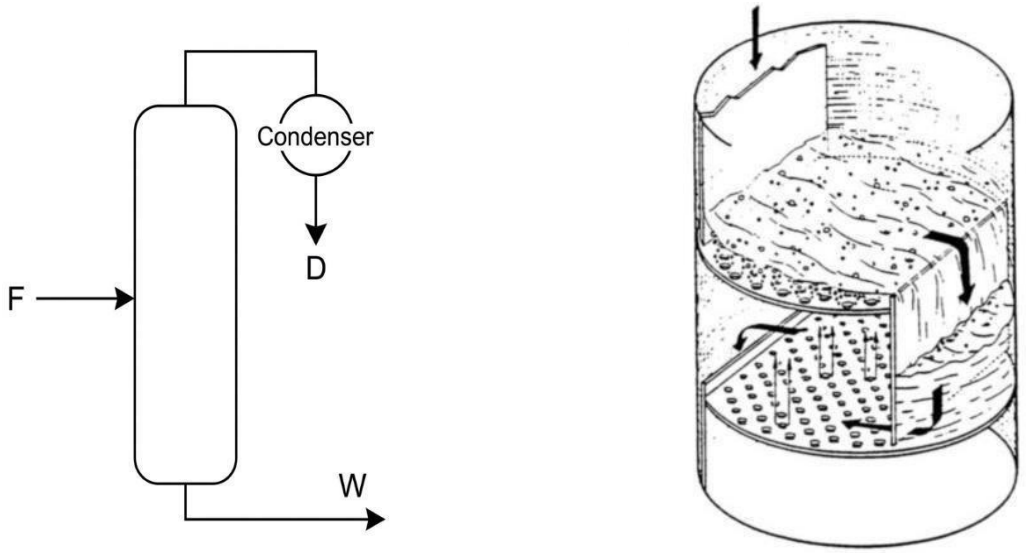

Fig. V.2. Flow patterns in a trayed column.

- **Packed columns**: these are columns in which the internal space is filled with packing materials designed to significantly increase the contact surface area between the liquid and the vapors. There are two main types of packing:

o Loose packing, consisting of a multitude of small elements.
o Structured packing.

The packing in a column is designed to bring the liquid phase, which flows downward by gravity, into contact with the vapor phase, which rises toward the top of the column.

- **Characteristics, Advantages, and Applications of Packed Column:**

• It must provide sufficient void space to allow the liquid and vapor/gas to flow in countercurrent without excessive pressure drop.

• It must also provide a large wetted surface area to ensure a significant mass transfer surface between the liquid and the vapor (transfer of volatile components from one phase to the other). For comparable sizes, a packed column is more efficient than a tray column, but it also clogs much more easily. Packed columns are generally are preferred over tray columns in the following cases:

• Columns with a small diameter (< 600 mm).

• When dealing with corrosive fluids requiring special metallurgical materials.

• When pressure drops must remain low, which is particularly the case for vacuum distillation columns.

• When the liquid flow rate is high enough to minimize distribution problems within the column.

• When the liquid contains no solid particles (risk of clogging the packing).

• When there is a risk of foaming: since contact between the liquid and gas phases is less violent, the tendency to foam is minimized.

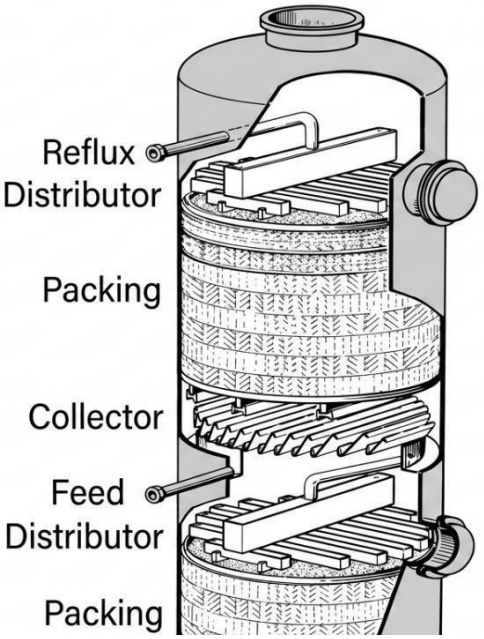

Fig. V.3. Internal components of a packed column.

V.4. Principle of distillation:

V.4.1. Liquid-vapor equilibrium:

Distillation involves separating a mixture of liquids by evaporating and condensing the vapors. The separation of compounds by distillation of an ideal mixture is based on Raoult's law: the vapor pressure $P_{(mix)}$ of an ideal solution is equal to the sum of the vapor pressures p_i of its components, proportional to their respective *molar* fractions x_i in the liquid phase:

Now, the partial pressure of a component in a mixture is proportional to the molar fraction of that component in the gas phase and to the pressure of the mixture:

$$P_{mél} = \sum_i p_i \times x_i$$

Now, the partial pressure P_i of a component of a mixture is proportional to the molar fraction y_i of that component in the gas phase and to the pressure of the mixture:

$$P_i = y_i \times P_{mél}$$

$$P_i = y_i \times P_{mél} = p_i \times x_i$$

Hence

$$y_i = K_i \times x_i$$

Let:

For an ideal mixture, if the gas phase is in equilibrium with the liquid phase, then the molar fraction y_i of one of the mixture's components in the gas phase is proportional to the molar fraction x_i of that same component in the liquid phase.

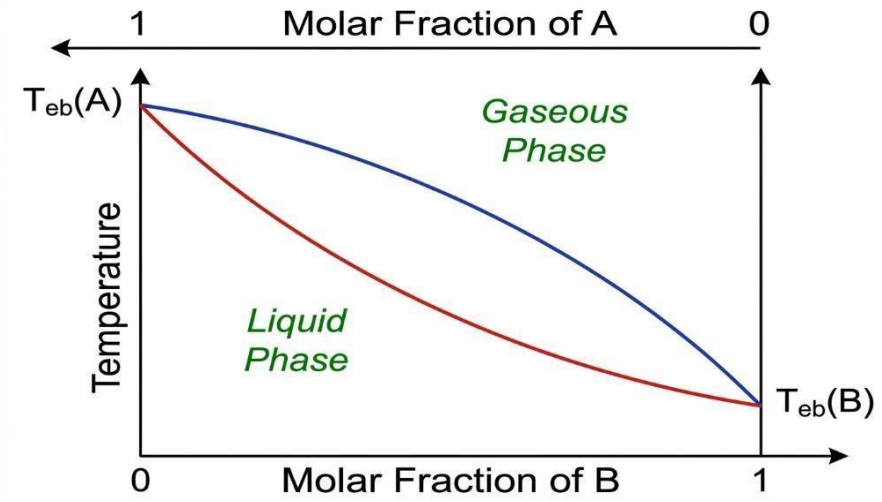

Fig. V.4. Phase diagram of a binary mixture.

For a binary mixture, the equilibrium constants can be determined graphically by plotting the dew line (in blue) and bubble line (in red) on the same graph (phase diagram). The graph below shows these lines plotted at constant pressure—which corresponds roughly to the conditions of conventional distillation. The binary mixture consists of compound A (heavy) and compound B (light).

The composition of the vapor in equilibrium with the liquid can be determined graphically:

1. We know the composition of the liquid (x_B), and since we are at liquid-vapor equilibrium, we must be on the bubble curve (red).

2. Since the vapor is in equilibrium, it is at the same temperature as the liquid: the equilibrium point is therefore on a horizontal line.

3. Since the vapor is in equilibrium with the liquid, we are on the dew line (blue); the vapor composition is therefore known (y_B).

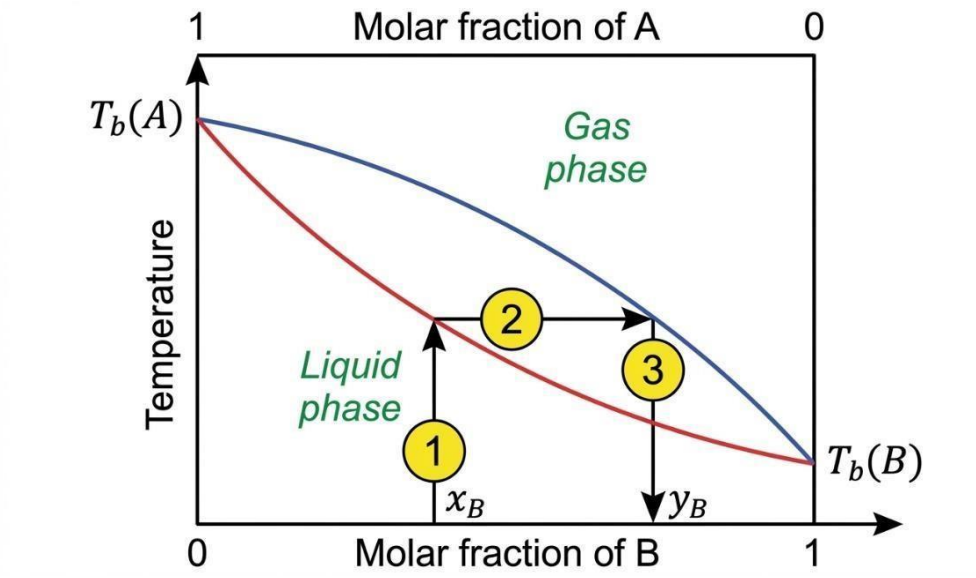

Fig. V.5. Determination of the composition of the vapor in equilibrium with the liquid.

The composition of the liquid in equilibrium with the vapor can be determined by proceeding in the reverse direction.

V.4.2. Batch distillation - simplified approach

Batch distillation involves evaporating the contents of a vessel and condensing the resulting vapors. The composition of the first drop of condensed vapor can be determined using the bubble and dew point curves.

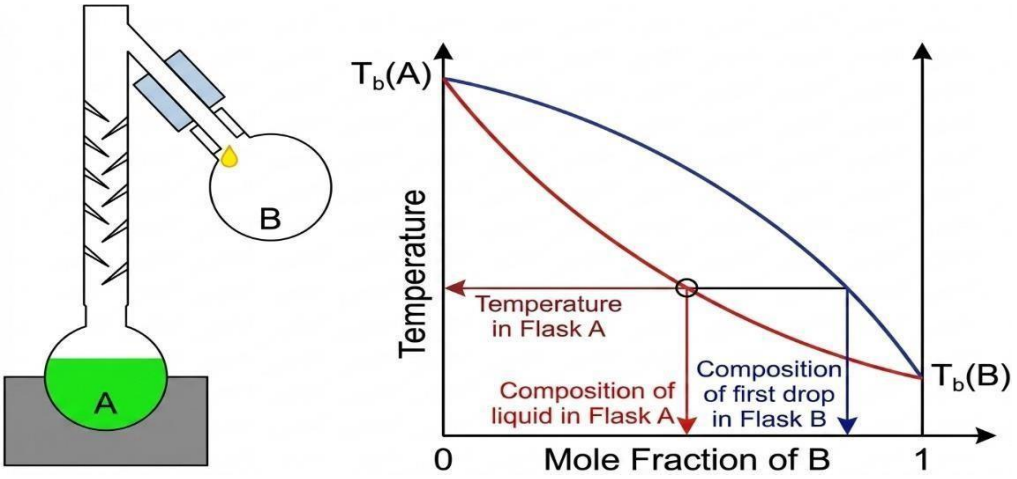

Fig. V.6. Composition of the first drop of liquid.

In the previous setup, with total condensation, the composition of flask B actually corresponds to the composition of the gas phase; the condensation of the vapor merely liquefies it.

The condensed vapors are richer in light components than the contents of the flask. As distillation continues, given the conservation of mass, the light fraction in the flask is depleted. This change in composition shifts the flask's operating point to the left. The flask remains in equilibrium with the vapor phase and thus remains on the bubble curve. This shift causes the temperature in flask A to rise.

We also observe a change in the composition of the gas phase, and consequently in the composition of the liquid in flask B, which will gradually become enriched in heavy components.

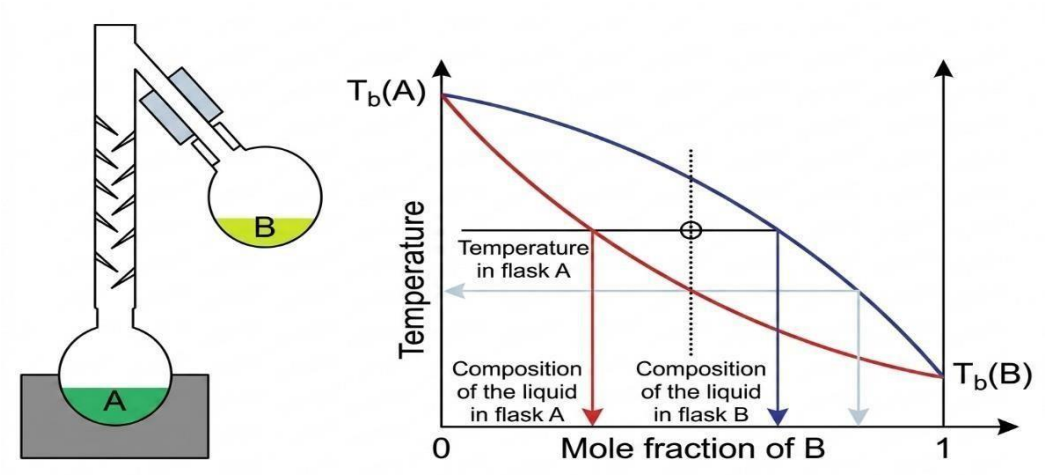

Fig. V.7. Continuation of distillation.

If distillation is continued until the last drop of liquid, its composition can be determined graphically from the dew point and bubble point curves. The temperature in the flask continues to rise.

Ultimately, flask B will contain the entire initial contents of flask A.

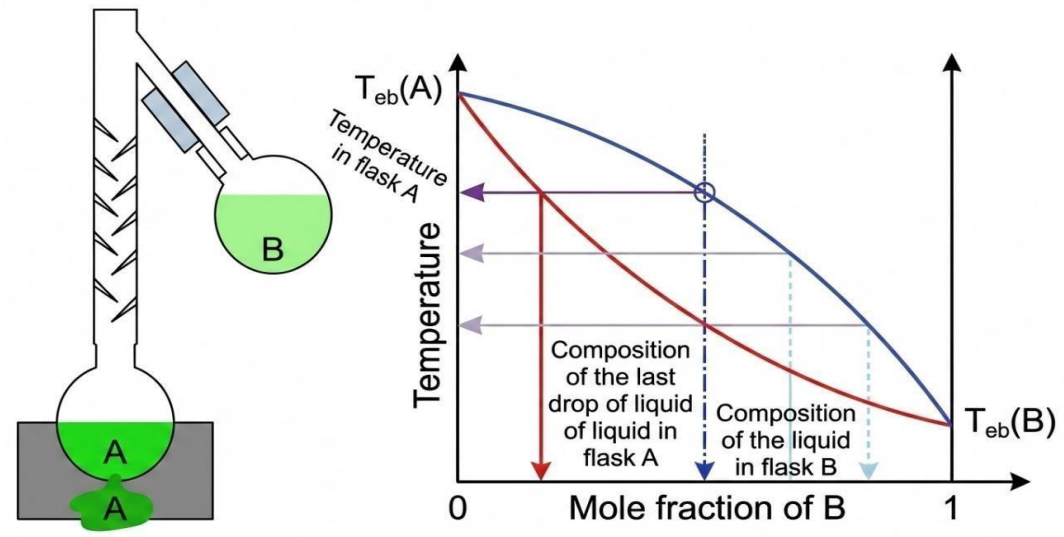

Fig. V.8. Composition of the last drop of liquid.

Limitations of Simple batch distillation in binary mixture separation:

- Simple batch distillation does not allow for the efficient separation of a binary mixture:

o The maximum molar fraction of the light compound corresponds to that of the first drop.

o The maximum molar fraction of the heavy compound corresponds to that of the last drop.

- If the ongoing distillation is stopped, two mixtures of different compositions can be separated: o The contents of flask A have become enriched in the heavy compound compared to the initial contents. This fraction is called the distillation **residue**.

o The contents of flask B are enriched in the heavy compound compared to the initial mixture. This fraction is called **the distillate**.

o To separate these two mixtures, further distillations would then be required.

It is explained in the figure V. 9 an example of purification by successive distillations.

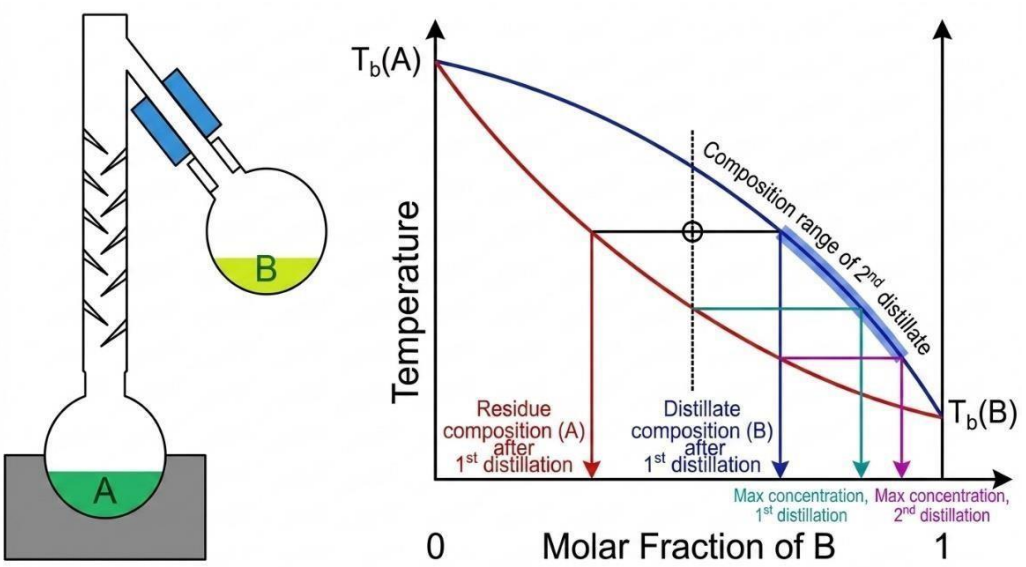

Fig. V.9. Example of purification by successive distillations.

V.4.3. Continuous fractional distillation - simplified approach

In batch distillation, the composition of the vapor changes over time due to changes in the composition of the feed. The feed composition changes because the lighter products "disappear" from the liquid phase and become concentrated in the vapor phase. In contrast continuous distillation...

Continuous distillation corrects this by continuously feeding the distillation process. As a result, a more efficient and stable separation is achieved compared to batch distillation.

Single-stage distillation

As a first approximation, continuous distillation can be likened to a two-phase separation. To simplify the explanation, we will consider a binary mixture of two components, A (the heavier one) and B:

- Knowing the composition and initial temperature of the feed (in the liquid phase), we can locate point A on the phase diagram of the binary mixture.
- The feed is heated to a temperature higher than the mixture's bubble point (point B, located vertically above A because the composition is identical).
- Upon reaching the flash drum, the liquid and gas phases separate. Assuming that thermodynamic equilibria have been reached:

o The composition of the gas phase, in equilibrium with the liquid, is read on the dew point curve.

o The composition of the liquid phase, in equilibrium with the vapor, is read on the bubble curve.

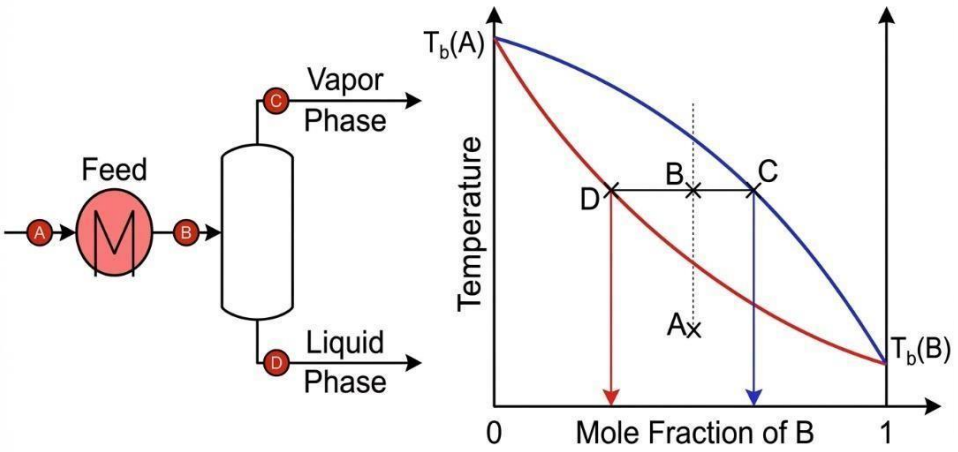

Fig. V.10. "Continuous distillation" with 1 stage.

The above setup thus allows the feedstock to be separated into two streams with fixed compositions:

- A gaseous phase, which can be condensed, enriched with light compounds relative to the feedstock (= distillate).
- A liquid phase, enriched in heavy compounds (= residue).

However, unlike in batch distillation, the compositions of the distillate and the residue are constant and depend only on the feed reheat temperature.

Note that the closer the heating temperature is to the bubble point, the more concentrated the distillate is in light products; but since less energy is "injected" into the feed, there is less evaporation and therefore the distillate flow rate is lower.

Three stage distillation

Separation can be improved by adding stages, and by:

• Partially cooling the gas-phase outlet of the first separator to a temperature of T2.

• Reheating the liquid-phase outlet of the first separator to a temperature T3.

This type of distillation is shown in the figure below.

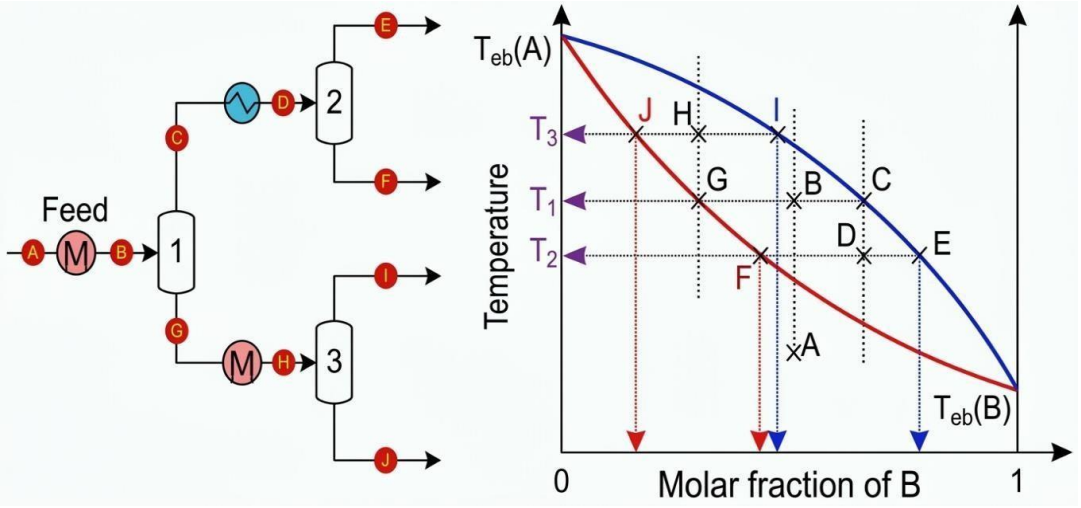

Fig. V.11. Multistage continuous distillation system with 3 stages.

This results in 4 streams (E, F, I, and J), including:

• A stream E more concentrated in light fractions than the feed, or than the gas outlet of the first separator (C).

• A stream J more concentrated in heavy fractions than the feed, or than the liquid outlet of the first separator (G).

However, there are also two streams, the F and I outlets, which here have a composition similar to that of the feed and which it might be economically advantageous to recycle into the first separator. This can be achieved using the following equivalent setup, by replacing the heaters and separators with stirred and heated reactors.

V.4.4. Fractional Distillation (Rectification or Distillation with Reflux)

To achieve advanced separations (obtaining components with high degrees of purity, particularly when these have similar volatilities), rectification is often used. Rectification is a technique that employs repeated distillation in a column to facilitate mass transfer between the rising vapor phase and the falling liquid phase within the column. Rectification consists of an exchange of material between a liquid phase and a vapor phase flowing in countercurrent within a column designed to bring the two phases into intimate contact. Although based, in part, on the same fundamental physical laws as simple or fractional distillation, it differs from them in an essential way.

If we return to the previous example and recycle the liquid condensed in the flash tanks located above the feed to the lower stages (and the vapor produced in the flash tanks located below the feed to the upper stages), we obtain the schematic diagram of the distillation process as illustrated in figures (V.12) and (V.13).

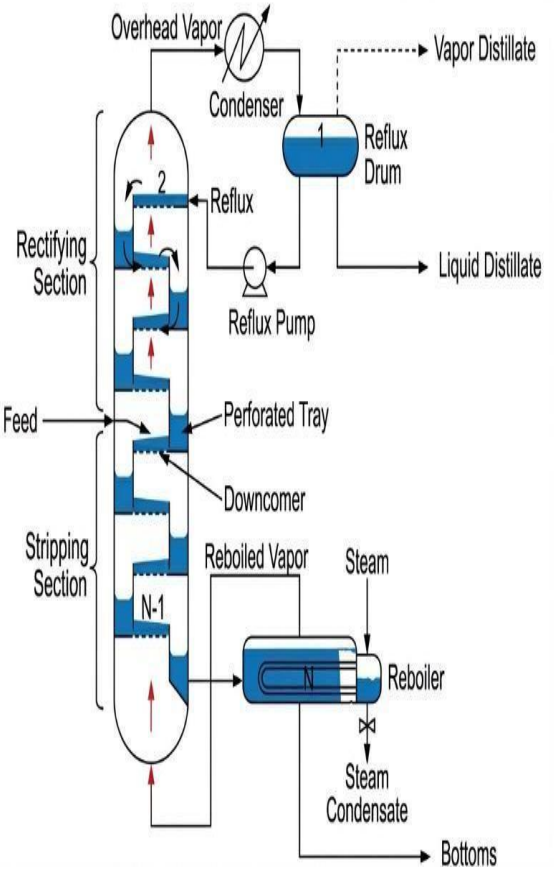

Fig. V.12. Schematic diagram of the fractional distillation process for the separation of a binary mixture (A–B).

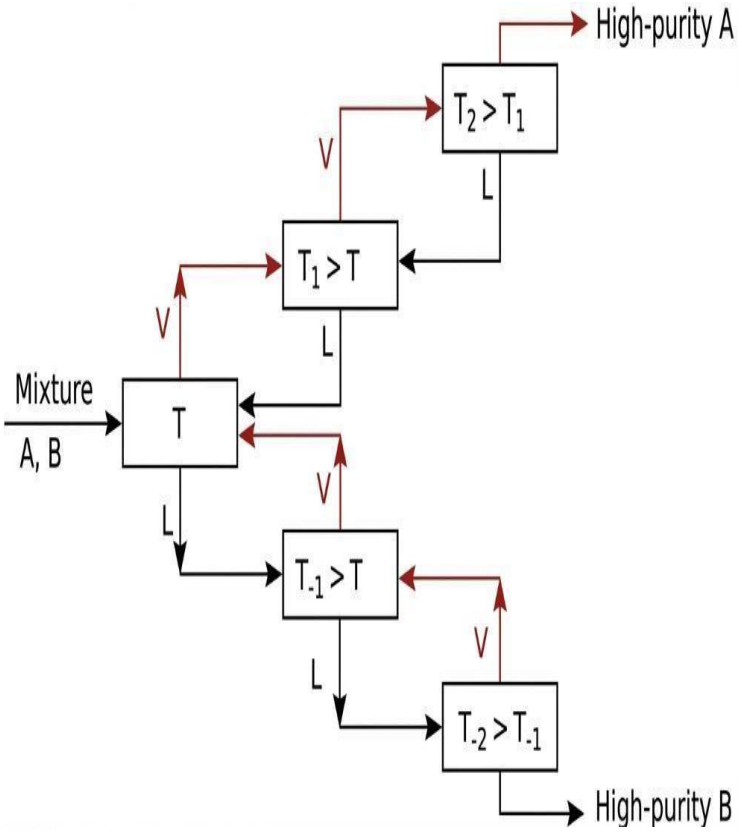

Fig. V.13. Practical of distillation column.

Distillation column

- In this example, we see that the selective separation of the components of a mixture relies on the existence of a temperature gradient between the bottom and the top of the column.
- Most often, this temperature gradient is maintained simply by cooling the top of the distillation column (condenser) and heating the bottom (reboiler).
- with the temperature gradation ensured by the mixing of fluids at each tray (vapor and liquid intersecting at each stage).
- Cooling the condenser creates a liquid phase that flows down the column, while heating the reboiler produces a vapor phase that rises up the column: this is therefore referred to as a countercurrent separation process.

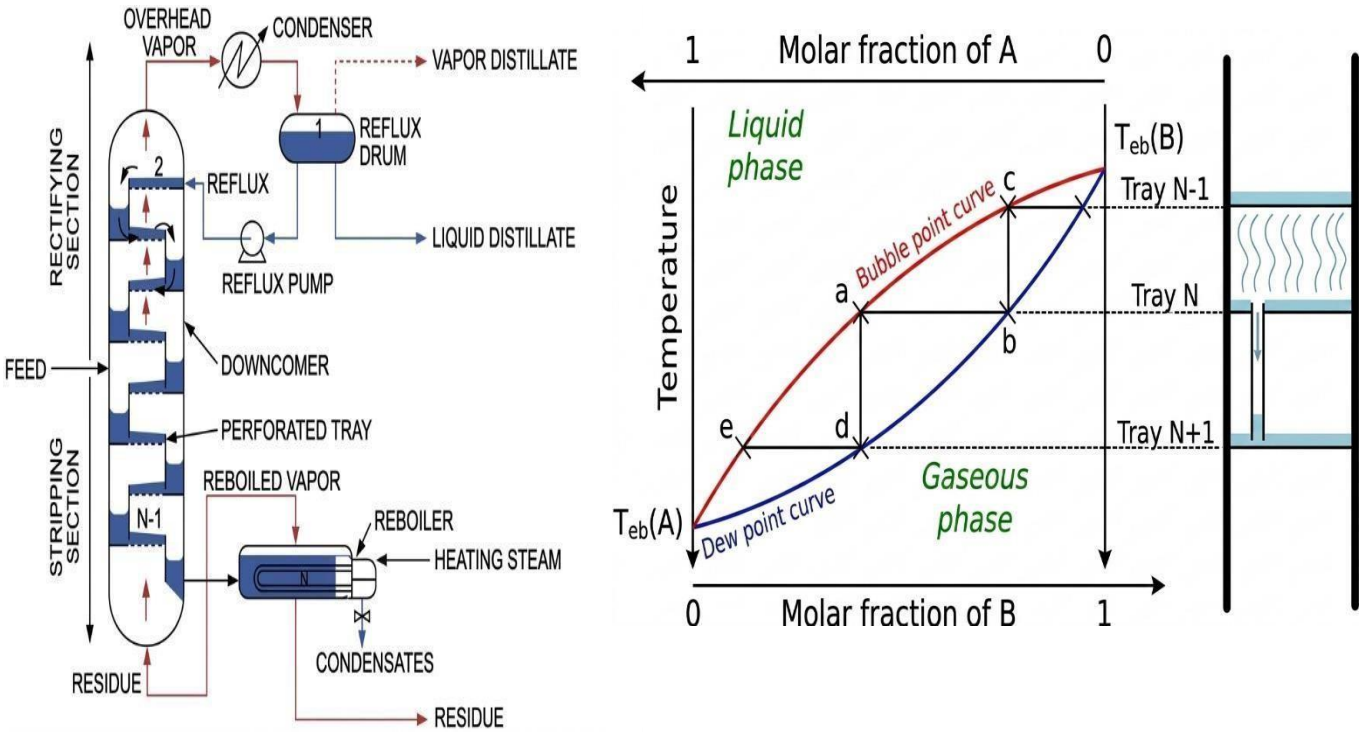

Fig. V.14. Fractional distillation: graphical determination of Liquid-Vapor equilibrium on trays.

The concept of **a theoretical plate** is defined as a plate located in an ideal column where:

- Thermodynamic equilibria are instantaneous.
- There is no loss of energy.

Consider the idealized N-phase system:

- Point a, located on the bubble curve, corresponds to the composition of the liquid in this tray.
- Point b, located on the dew point curve, corresponds to the composition of the vapor in equilibrium with the liquid.

We can clearly see that the vapor is richer in compound B (light) than the liquid from which it originated.

Now consider tray N-1, also assumed to be ideal. In a fully refluxed column at equilibrium, the composition of the liquid is identical to that of the gas (see Fig. V. 14). The operating point representing the liquid is therefore located vertically above that of the gas (b), and since we are in the liquid phase, in equilibrium with its gaseous phase, it is also on the bubble curve of the mixture (it is therefore indeed point c).

We clearly observe a decrease in temperature as we move up the column, as well as a gradual enrichment in the lighter compound (here, B).

The reverse is also true as you move down the column.

- **The position of the feed indicates the role of the column**

  - ***Feed in the high position***

    - No more trays in the discharge zone;
    - The focus is on the quality of the residue.

  - ***Feed in the lower position***

    - More trays in the rectification zone;
    - The quality of the distillate is what is sought.

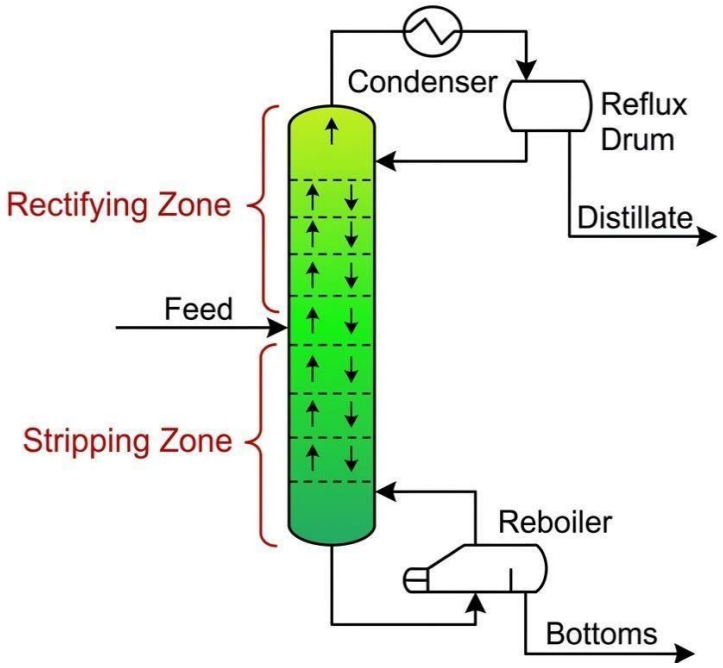

Fig V. 15. The position of the feed.

V.5. Calculation of the efficiency of a rectification column

V.5.1. Graphical methods of Mac Cabe and Thièle

The McCabe and Thiele models are derived from the mass balance equations for each tray or section of a distillation column, based on the following assumptions:

- The latent heats of vaporization of the two components are identical;
- The column is adiabatic;
- The condensation of the top-of-column vapors is complete.

They allow us to determine, for a given separation (x_f, x_w, and x_d):

- The minimum number of theoretical stages (NET_{min} at full reflux);
- The minimum reflux ratio (R_{min} at infinite number of plates);
- The number of theoretical stages NET required for a reflux ratio R.

- The number of actual trays or the required packing height;

  The reflux ratio R is generally chosen such that $1.2\ R_{min} < R < 2\ R_{min}$. It represents a compromise between:

- The number of trays (capital cost)
- The reflux ratio (associated energy cost, i.e., operating cost).

Tray efficiency: this represents the difference between a theoretical tray (phases leaving the tray in liquid-vapor equilibrium) and an actual tray. The relationship is expressed as:

NET-1 = Number of actual trays × tray efficiency

Height Equivalent to a Theoretical Plate (HEPT): This is the required packing height (in meters) to achieve liquid-vapor equilibrium, or a theoretical plate, in the column. The relationship between packing height and HEPT is expressed as follows, for NET-1 theoretical plates: ***Packing height = HEPT × (NET-1)***

The actual number of trays or the packing height of a column is related to NET-1 (Number of Theoretical Stages minus the reboiler), as determined by the McCabe and Thiele equations, because the reboiler is always considered a theoretical stage—that is, it establishes a liquid-vapor equilibrium.

When analyzing a column in operation, we determine:

Tray efficiency = (NET-1) / Number of actual trays

HEPT = Packing height / (NET-1)

When sizing a system, the following are determined:

Number of actual trays to be installed = (NET-1) / tray efficiency

Packing height to be installed = (NET-1) × HEPT (HEPT = Height Equivalent to a Theoretical Tray, provided by the packing manufacturer).

The tray efficiency or HEPT is calculated separately or provided by the equipment manufacturer. Their values depend on the properties of the fluids under operating conditions and the operating conditions of the column.

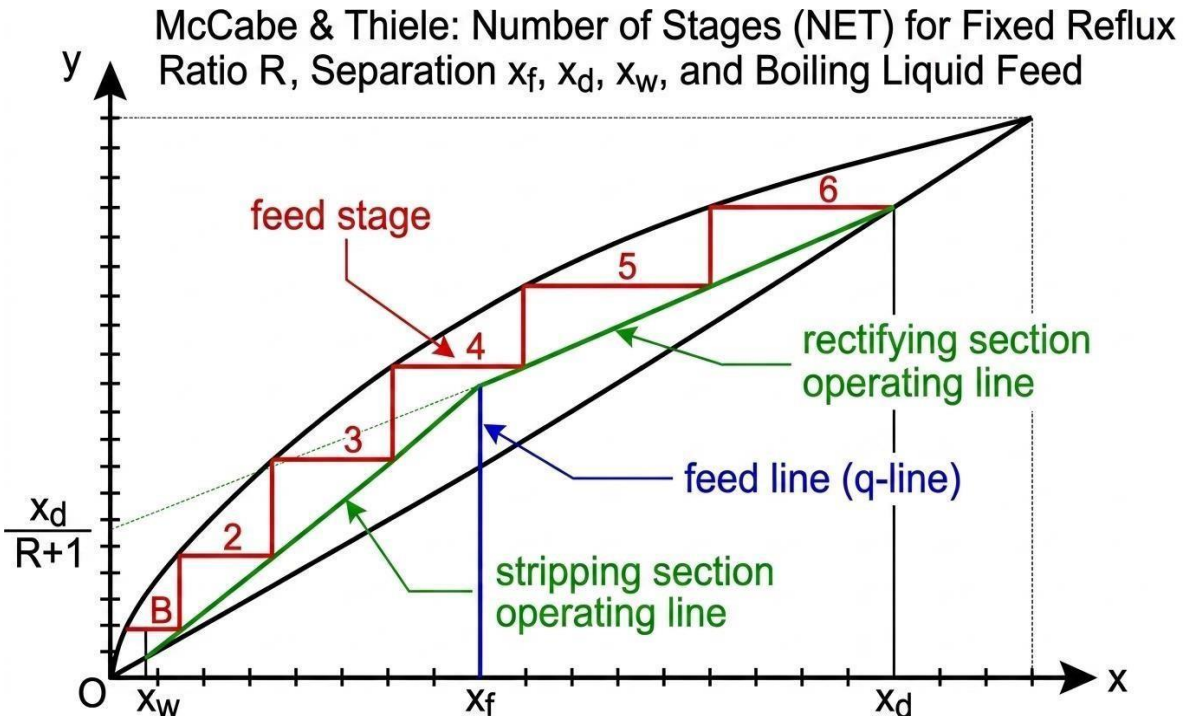

Fig. V.16. Graphical construction and determination of the number of trays using the MacCabe and Thièle method.

V.5.2. Ponchon–Savarit graphical method:

This method requires knowledge of the enthalpy-composition diagram; its advantage is that it determines the number of trays, the compositions, temperatures, and enthalpies of the different phases at each stage, without taking into account Lewis's assumptions, unlike the McCabe and Thièle method, which requires only knowledge of the (y, x) diagram.

Enthalpy-concentration diagram: The molar fractions x (liquid) or y (vapor) are plotted on the x-axis, and the y-axis represents enthalpy. The h_L and H_V curves represent the enthalpies of a liquid and a saturated vapor, respectively.

The lower and upper parts of the graph correspond to the liquid and vapor phase regions. Between the two curves, both liquid and vapor phases exist, whose equilibrium compositions at a given temperature lie at the ends of a conoidal (or conjugate line). The isotherms in the single-phase liquid or vapor zones indicate the enthalpy level of a subcooled liquid or a superheated vapor. The use of the inverse lever arm rule allows for the calculation of the respective quantities of each phase in a liquid-vapor mixture.

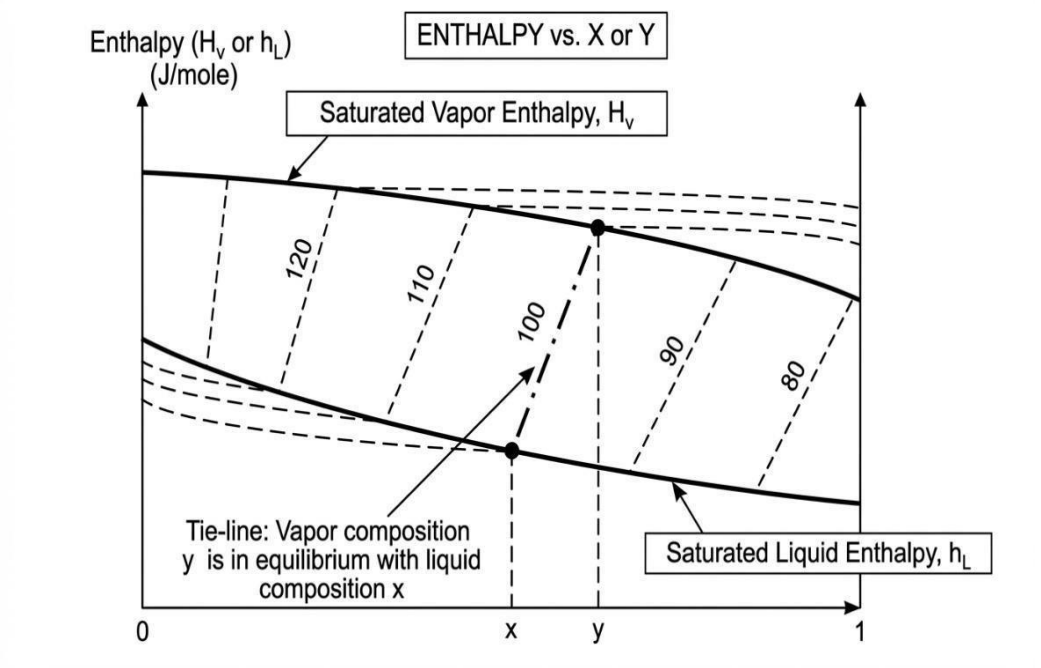

Fig. V.17. Enthalpy-concentration diagram.

V.6.Vacuum distillation

Distillation is a process that can be carried out under a wide range of pressures. It is performed under pressure when the feedstock is highly volatile. Increasing the pressure raises the dew point, allowing the vapor to be condensed by water. Vacuum distillation is used when distilling a mixture containing either very low-volatility products or thermally unstable products.

- Vacuum distillation is a distillation process carried out at a pressure lower than atmospheric pressure, causing the evaporation of the most volatile liquids at reduced temperatures.
- It is reserved for the fractionation of products whose boiling point at atmospheric pressure would be too high and would lead to cracking of the feedstock (degradation of the product)

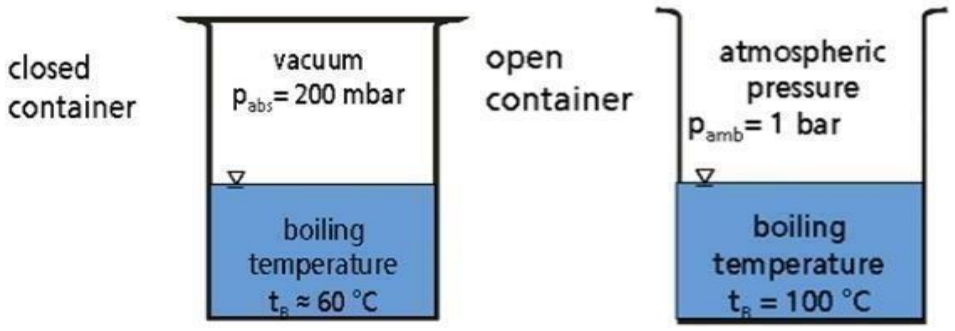

Fig. V.18. Vacuum distillation.

V.7. Azeotropic distillation

This type of distillation is specifically used for azeotropic mixtures.

- They cannot be distilled directly because the liquid composition and the vapor composition are identical.
- In this case, another component is added to create a new low-boiling-point azeotrope, thereby facilitating separation.
- In ethanol production, benzene is added to break the azeotrope of the mixture of 96% ethanol and 4% water.

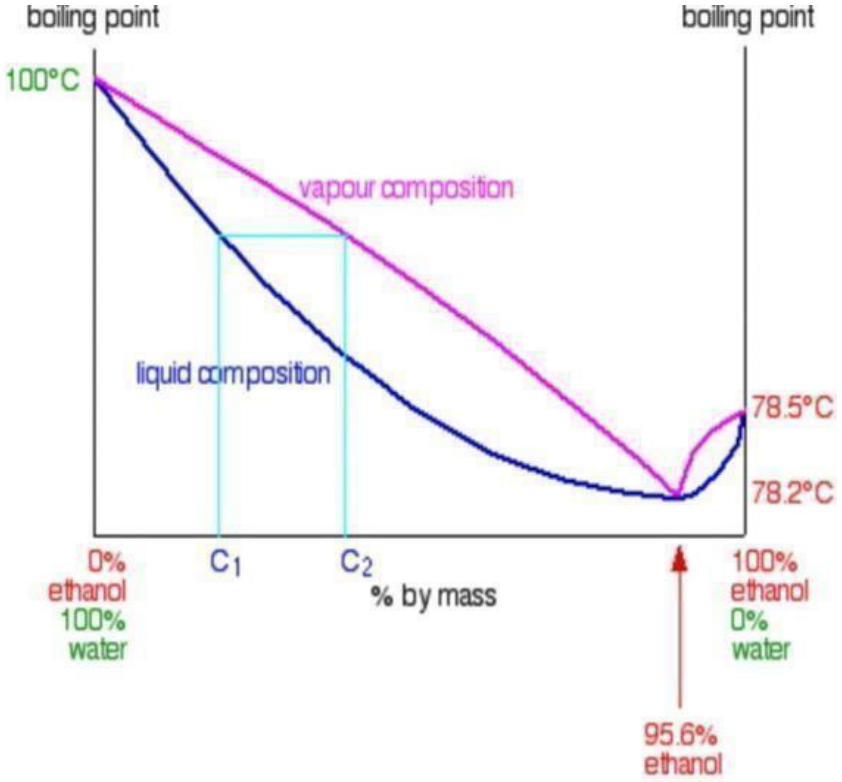

Fig. V. 29. Azeotropic distillation.

V.10. Application exercises:

Exercise 1:

We wish to continuously distill a mixture of **2000 kg/h** containing **60%** methanol in water to obtain a distillate containing **98%** methanol and a residue containing **95%** water (by mass).

Calculate the mass flow rates of the distillate and the residue.

Exercise 2

A distillation column for a mixture of LPG 'Liquified petrol gas' with the following molar composition:

Table. V. 1. Molar composition of LPG.

Components	(%) molar
CH_4	0.4
C_2H_6	1
C_3H_8	36
C_4H_{10}	41
C_5H_{12}	21.6

Values set by the process:

The feed rate F = 22,000 kg/h

The C4 content in the distillate is 1.5%

The C3 content in the residue is 5%

1. Calculate the mass composition, mass flow rates, and molar flow rates in the feed for each component;
2. Calculate the compositions and flow rates (mass and molar) of each compound in the distillate; calculate the molecular mass of the distillate;
3. Calculate the compositions and flow rates (mass and molar) of each compound in the residue; calculate the molar mass of the residue.

Exercise 3

The equilibrium curve of the ethane–propane mixture (assumed to be ideal) at 13.55 atm is given below. A vapor mixture with a molar composition of 65% ethane and 35% propane is considered. It is desired to condense one-half of this vapor (on a molar basis) (see Fig. V. 21).

What will be the compositions of the two phases thus formed?

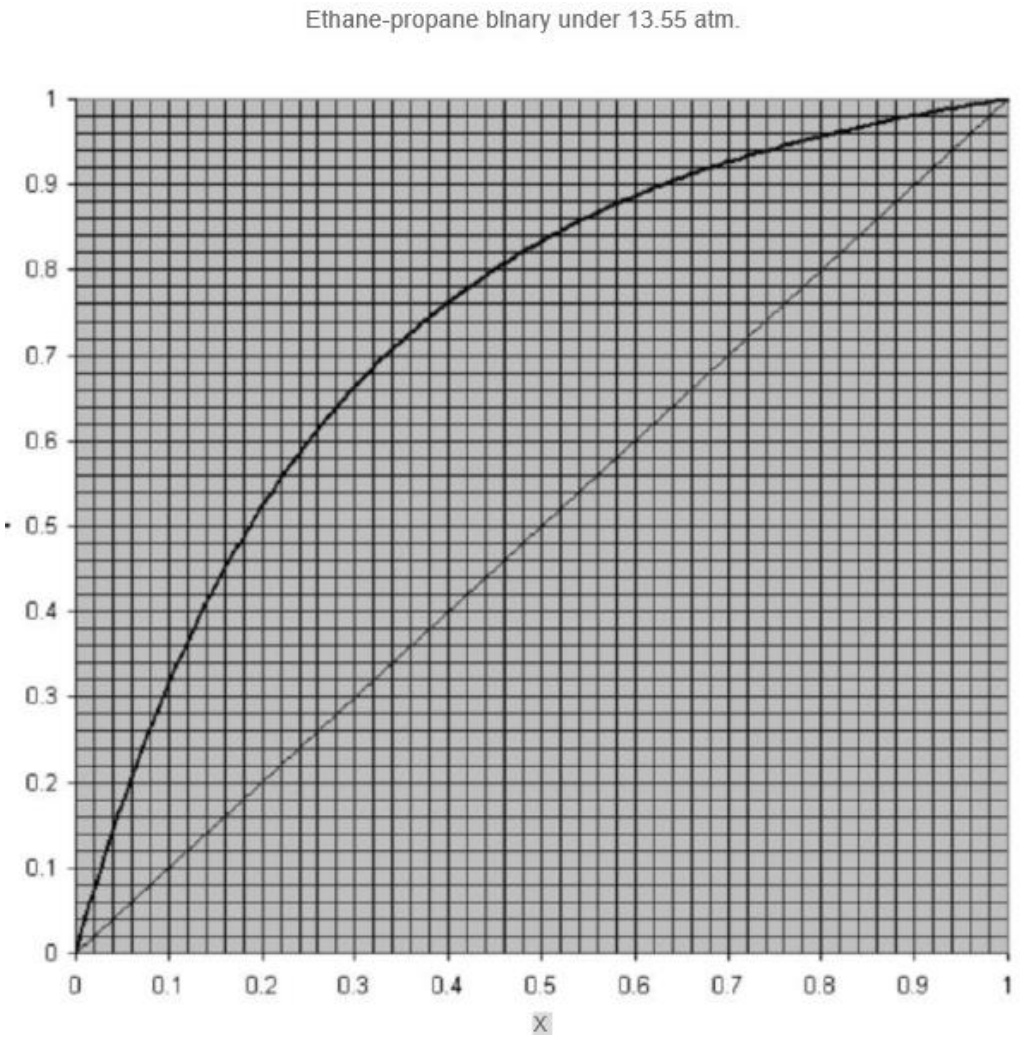

Fig. V. 21. Equilibrium curve of the ethane–propane mixture.

Exercise 4

A boiling liquid mixture of toluene and xylene with a mole fraction of 0.55 toluene is fed to a continuous rectification column at a flow rate of F = 2000 mol·h⁻¹. The objective is to obtain a distillate at the top of the column with a mole fraction of x_D = 95% toluene and a bottom product with a mole fraction of x_B = 5% toluene.

1) Draw the detailed process flow diagram of the continuous rectification system.

2) Determine all flow rates as well as the mass and molar compositions of the streams, and present the results in a table.

3) Determine the minimum number of theoretical stages required to achieve the separation under total reflux conditions.

4) Determine the minimum reflux ratio required to achieve the specified separation.

5) For a reflux ratio equal to 2, determine the number of theoretical stages required. The feed stage is represented by a vertical line.

The stages should be numbered from bottom to top, with the reboiler considered as Stage No. 1.

Given data:

Molecular weight of xylene: $M_{xylene} = 106\ g \cdot mol^{-1}$

Molecular weight of toluene: $M_{toluene} = 92\ g \cdot mol^{-1}$.

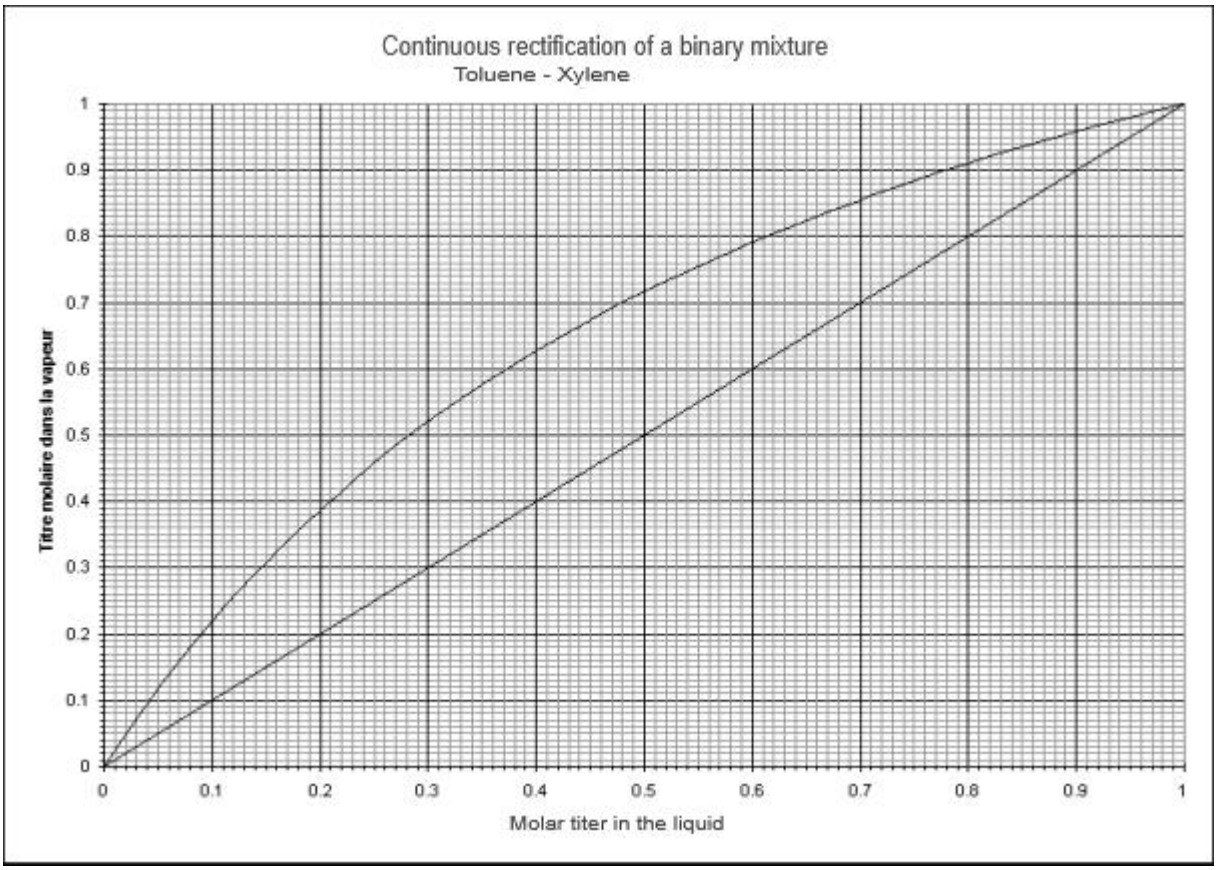

Fig. V. 22. Vapor–liquid equilibrium (VLE) curve for the Toluene–Xylene system.

Exercise 5

The rectification of a binary mixture (A + B) with a feed composition of z_F = 40 mol% of the more volatile component (A) produces a distillate containing x_D = 90 mol% (A) and a bottoms product containing x_B = 93 mol% (B) (mole fractions).

The feed is introduced as a saturated vapor (at its dew point). The absolute volatilities of A and B are 5.7 and 2, respectively. The feed flow rate is 1000 $kg \cdot h^{-1}$.

The vapor and liquid phases may be assumed to be ideal, and the gases are considered perfect gases. **Total condensation** is employed. The reference temperature is **25 °C**.

Given:

Using **exclusively the Ponchon–Savarit method**:

a) Determine the **minimum reflux ratio** by taking as a basis the **tie-line passing through point F** (corresponding to the feed).

b) If a reflux ratio **R** equal to **75% greater than the minimum reflux ratio (Rmin)** is used, determine the **number of theoretical stages (NTS)** required for this rectification.

- On which stage should the feed be introduced?

c) Determine the heat duties involved at the **reboiler (Q_B)** and the **condenser (Q_C)**.

d) Calculate the liquid flow rate **L_2** descending from the **second tray from the top** of the column.

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