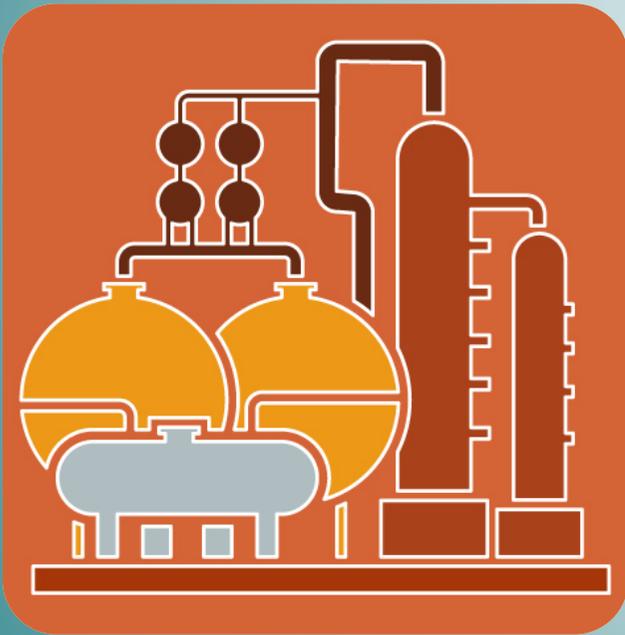


Chemical Process Design / Diseño de Procesos Químicos

Topic 7. Process synthesis: distillation sequences



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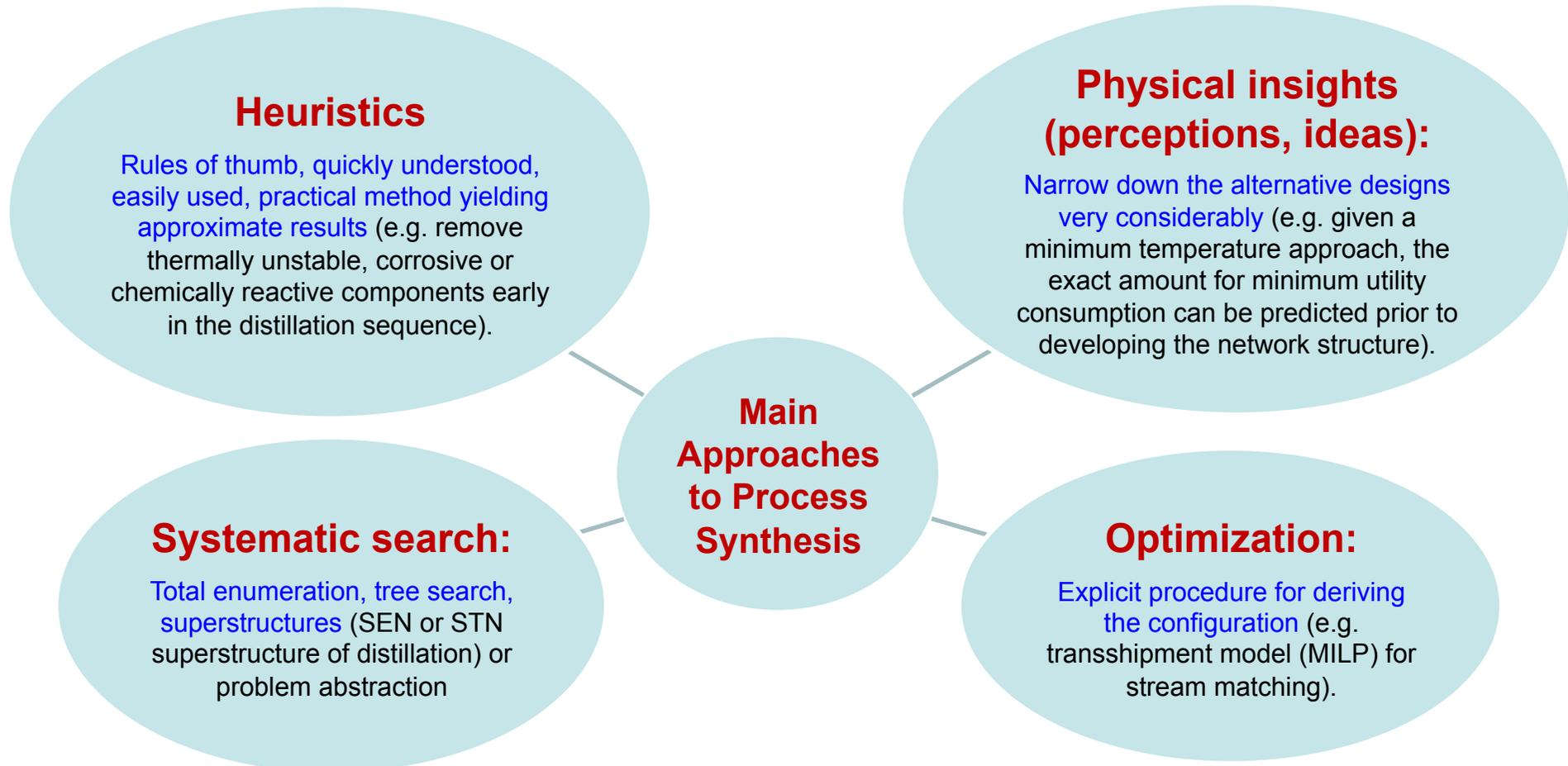
4.- Further Reading and References

RELEVANT TO LEARNING

1.- Process synthesis



How to derive “optimal” configuration of a process or subsystem
→ Topology \equiv Which Units?, How to interconnect?



2.- Synthesis of distillation sequences

- **Given multicomponent feed separate into N high priority products. Assume: Ideal behavior, Splits near 100% recovery.**

- 3 components: A, B, C → 2 sequences (direct, indirect) (*)
- 4 components: A, B, C, D → 5 sequences (*)
- Increase n° components → Increase n° sequences →

$$\rightarrow \text{N° flowsheets} = [2 (N - 1)! / [(N - 1)! (N)!]$$

- **Other alternatives:**

- **Petlyuk column** (1 condenser, 1 reboiler, lowest level of energy, most energy efficient) (*)
- **Side Stream Columns** (1 condenser, 2 reboilers) (*)
- **Extractive Distillation** (*)

- **If several technologies exist for separation (adsorption, extraction, other distillations), there are more alternatives.**

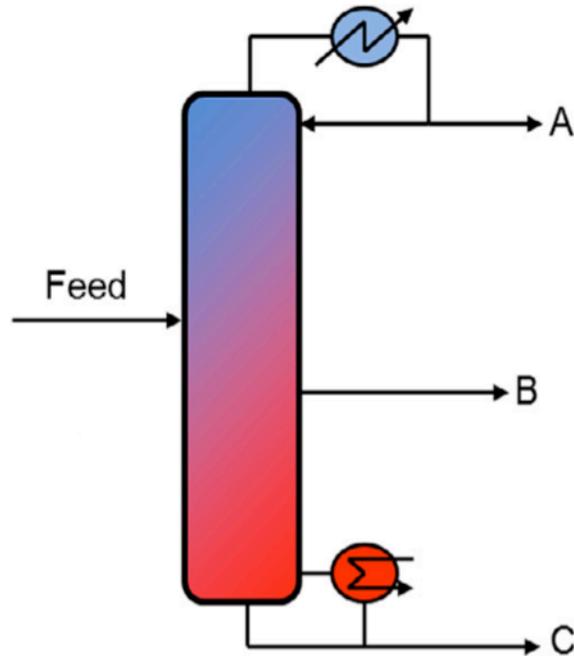
Distillation → 14 flowsheets

N = 5 →

$$\rightarrow \text{N° flowsheets} = \{[2 (N - 1)! / [(N - 1)! (N)!]\} S^{N-1}$$

3 technologies (S) → 1134 flowsheets.

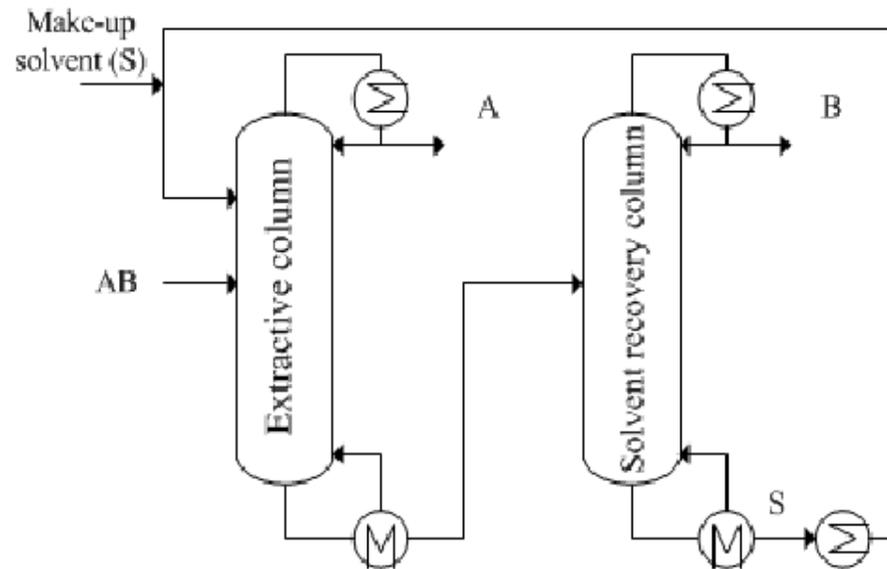
Side Stream Column



Long, N.V.D.; Minh, L.Q.; Nhien, L.C. & Lee, M. (2015):
«A novel self-heat recuperative dividing wall column to
maximize energy efficiency and column throughput in
retrofitting and debottlenecking of a side stream column».
Applied Energy, 159. Pp. 28-38.

- **Offer a cost-effective way of producing three products from a single column:**
 - Limited purity of the intermediate component product stream is a problem.
 - A high-purity side stream might require large reflux ratios and a large number of stages, as well as larger associated energy requirements (Long *et al.*, 2015).

Extractive Distillation

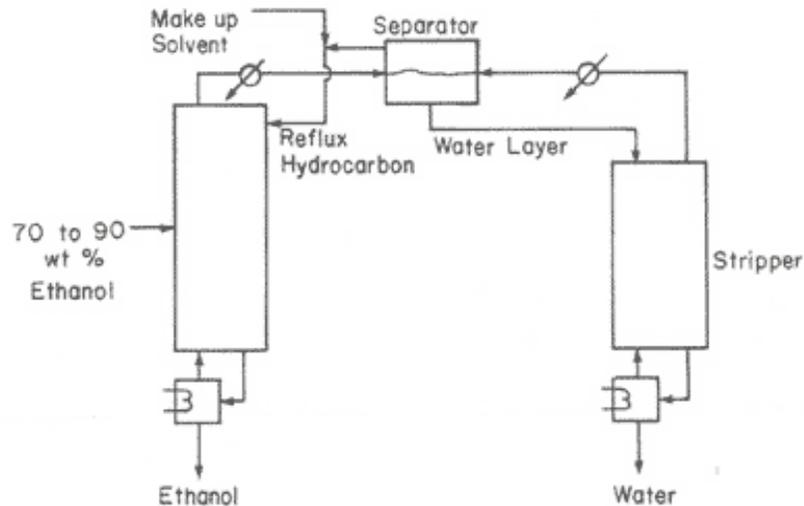


Nhien (*et al.*) (2014): «Application of mechanical vapor recompression to acetone-methanol separation». International Journal of Chemical Engineering and Applications, 5, 3 (2015-2018).

- **Extractive distillation (ED) is used in the industry for the separation of mixtures with similar relative volatilities and azeotropes, for example the separation of:**
 - Hydrocarbons with close boiling points.
 - The recovery of aromas or fragrances.
 - Aqueous alcohol solutions.
 - Ether and alcohol mixtures.
 - Methylal and methanol mixtures.

Anokhina, E. & Timoshenko, A. (2015): «Criterion of the energy effectiveness of extractive distillation in the partially thermally coupled columns». Chemical Engineering Research and Design, 99. Pp. 165-175.

Azeotropic Distillation



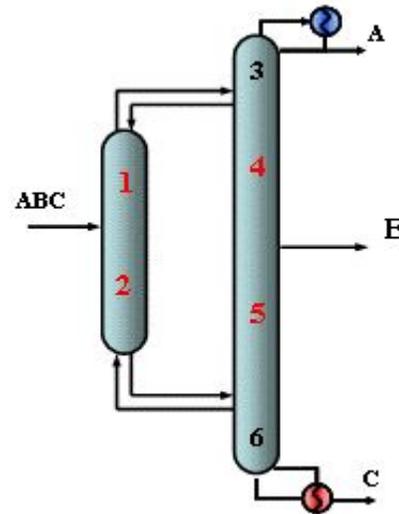
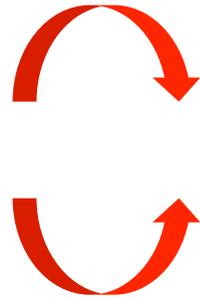
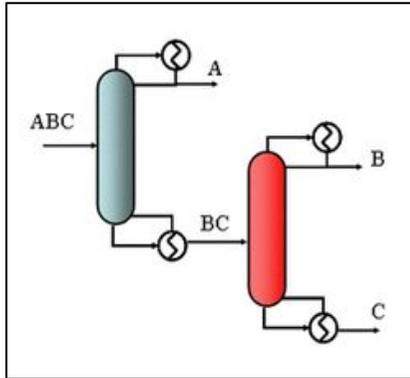
<http://www.hyper-tyt.ethz.ch/distillation-azeotropic.php>

The ethanol-water mixture can be broken using Pentane as the entrainer. This produces the formation of a heterogeneous ternary azeotrope.

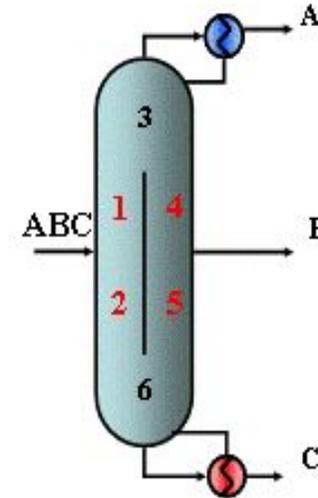
- **Azeotropic distillation is a method used to modify the equilibrium of complex mixtures in order to separate their components.**
- **Usually two different kinds of azeotropic distillation are distinguished:**
 - Binary systems which form a heterogeneous azeotrope.
 - Binary systems which form a homogeneous azeotrope. In this case an entrainer or solvent is added in order to form an azeotrope with one or both of the components. The system then becomes ternary.

2.- Synthesis of distillation sequences

Petlyuk column



Dividing wall column



http://iq.ua.es/~jose/DestilacionAcoplamientoTermico/Separacion_tres_componentes.html

Petlyuk column

The Petlyuk arrangement consists of a pre-fractionator coupled to the main column, using two recycle streams.

Dividing wall columns (DWCs)

Split the middle section of a single vessel into two sections by inserting a vertical wall. DWCs represent a typical example of process intensification since they can bring significant reductions in both capital investment and energy costs of up to 30%.

2.- Synthesis of distillation sequences

General Heuristics for Distillation

- Remove the most corrosive components first.
- Remove products as distillate.

Heuristics for sequencing Distillation trains (priority order)

- **H1:** forbidden splits:
 - a) Don't use distillation if $\alpha_{lk/hk} < 1.05$
 - b) If $(\alpha - 1)_{\text{extractive distillation}} / (\alpha - 1)_{\text{regular distillation}} < 5 \rightarrow$ Use ordinary distillation.
 - c) If $(\alpha - 1)_{\text{I-I extraction}} / (\alpha - 1)_{\text{regular distillation}} < 12 \rightarrow$ Use ordinary distillation.
 - d) Consider absorption if refrigeration is needed.
- **H2:** use the next separation of components that have the highest $\alpha_{lk/hk} \rightarrow$ easiest first, most difficult last
- **H3:** remove the most abundant component first
- **H4:** if α 's or concentrations are not very different \rightarrow Direct sequence
- **H5:** remove mass separation agent in next column
- **H6:** favor sequences that do not "Break" desired products

Apply rules in Decreasing Order of Priority.

3.- Examples of distillation synthesis (Practical Chapter)

- a) Demonstration of H4 rule:** if α 's or concentrations are not very different \rightarrow Direct sequence.
- Assume A, B, C equimolar mixture, feed liquid and similar $\alpha_{L/K}$
- b) Application of Heuristics. Problem 18.10 (Biegler, Grossmann and Westerberg, 1997):** using the heuristics, propose separation sequences for the following problem.
- Separate a mixture of six components *ABCDEF* into products *A*, *BDE*, *C*, and *F*.
 - Use either of two methods in developing your sequences
 - Distillation, method I. Component volatility order *ABCDEF*.
 - Extractive distillation, method II. Component volatility order *ACBDEF*.
 - Component amount (kmols/hr). *A*: 4.55, *B*: 45.5, *C*: 155.0, *D*: 48.2, *E*: 36.8 and *F*: 18.2.
 - Relative volatilities of the key species:
 - Method m I: *A/B*: 2.45, *B/C*: 1.55, *C/D*: 1.03, *E/F*: 2.50.
 - Method m II: *C/B*: 1.17, *C/D*: 1.70.
- c) Analysis of the Petlyuk Column (Energy Integration).**

4.- Further Reading and References

- Biegler, L.; Grossmann, I. & Westerberg, A. (1997): «*Systematic methods of chemical process design*». Prentice Hall.
- Doherty, M. & Malone, M. (2003): «*Conceptual design of distillation systems*». 1st Ed. Tata McGraw-Hill Education.
- Douglas, J.M. (1988): «*Conceptual design of chemical processes*». McGraw-Hill.
- Peter, M.; Timmerhaus, K. & West, R. (2005): «*Plant design and economics for chemical engineers*». 5^a Ed. McGraw-Hill.
- Seader, J.D. & Westerberg, A.W. (1977): «*A combined heuristic and evolutionary strategy for synthesis of simple separation sequences*». AIChE Journal, 23. P. 951.

RELEVANT TO LEARNING

- **To distinguish between the different Approaches to derive “optimal” configuration of a process or subsystem.**
- **Superstructures of distillation.**
- **Application of Heuristics.**