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REGSOlexpert : Entrainer Selection Tool for Waste Solvent Recovery by Batch Distillation Processes

RODRIGUEZ DONIS Ivonne, GERBAUD Vincent, BAUDOUIN Olivier, JOULIA Xavier

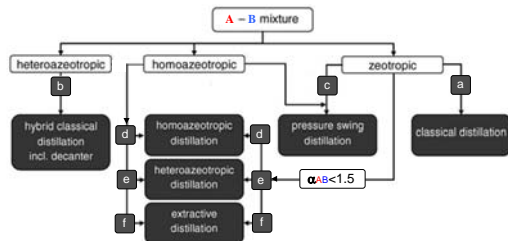
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Objective: General procedure to systematize the search of several alternatives enabling the separation of non-ideal binary mixtures such as pressure-swing distillation, azeotropic and extractive distillation.

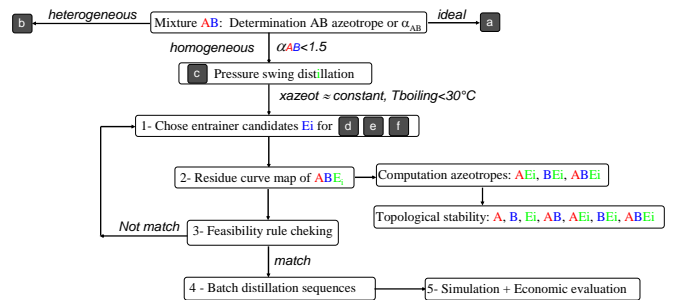
REGSOlexpert

- ✓ A wizard computer tool including 224 feasibility rules, and 326 batch azeotropic and extractive distillation processes.
- ✓ Systematic checking for each entrainer candidate for determining its feasibility to be used in rectifying or stripping batch column.
- ✓ A list of potential homogeneous and heterogeneous entrainers **E** in an optimal time for separating **A** and **B** as any industrial mixture.

Strategy for solvent recovery by distillation

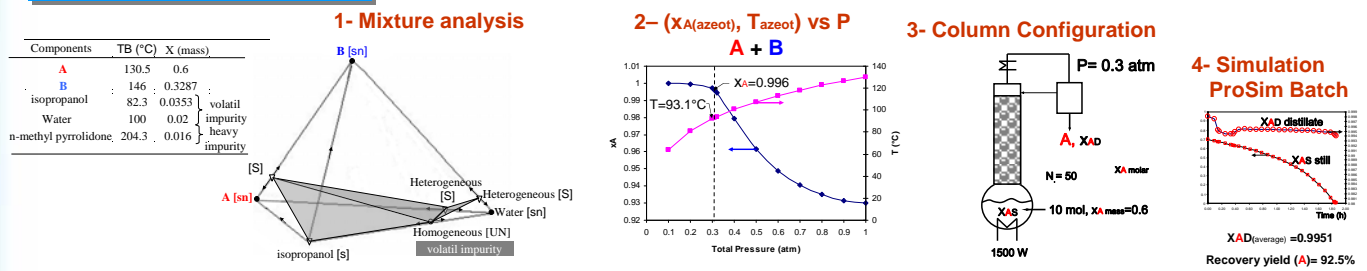


REGSOlexpert software algorithm



Industrial Applications

Pressure Swing Distillation Brabant Industry case: **Cyclopentanone (130.6°C)** – **Propylene Glycol Monomethyl ether (146°C)**



Extractive Distillation (HEBD)

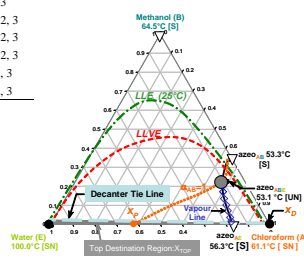
Chloroform (A) 61.1°C + Methanol (B) 64.5°C + Water (E) 100°C

USE: Extraction of bioactive substances from biological sources
TMIN Azeotrope: T = 53.5 °C, X_A = 0.65, γ_A[∞]/γ_B[∞] = 0.19

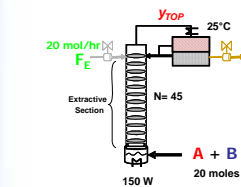
1 - Selecting Entrainer with RegSolExpert®

Entrainer	T _B (°C)	γ _A [∞] /γ _B [∞]	Process	Rejection
Acetic acid	118	1.87	BED	1, 3
Allyl alcohol	97	1.23	BED	1, 2, 3
Aliphatic Alcohols	96 - 118	≠1.50	BED	3
Water	100	398	HBED	-
n-Butyl acetate	126.5	0.11	BED	3
Furfural	161.4	0.42	BED	1, 2, 3
Pyridine	115.2	0.52	BED	1, 2, 3
Nitrobenzene	210.6	0.05	BED	1, 2, 3
4-methyl-2-pentanone	116.7	0.18	BED	2, 3
Methyl cyclohexane	101	0.02	HBED	2, 3

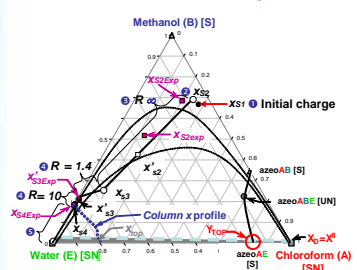
2 - Residue Curve Map



3 - Column configuration



4 - Simulation vs Experiments



- heat up F_E = 0 12 min
- R = ∞ F_E = 0 120 min
- R = ∞ F_E = 20 100 min
- R = 1.4 (D) F_E = 20 60 min
- R = 10 (-D) F_E = 20 20 min
- E-B separation F_E = 0 R = 1.5 250 min

excellent agreement

	purity	recovery
Water	0.9957	0.9690
Chloroform	0.9906	0.9914
Methanol	0.9988	-

Heterogeneous Azeotropic Distillation (HABD)

Acetonitrile (A) 81°C + Water (B) 100°C + Heterogeneous (E)

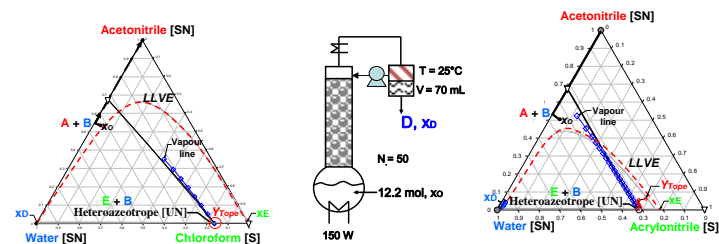
USE: Liquid chromatography separation in pharmaceutical Industry
TMIN Azeotrope: T = 77 °C, X_A = 0.67

1 - Selecting Heterogeneous Entrainer with RegSolExpert®

Initial List: 55 candidates from several chemical families (DIPPR database)

Heterogeneous E: Acrylonitrile and Chloroform form unstable heteroazeotrope with H₂O

2 - Residue Curve Map and 3 - Column configuration



4 - Simulation vs Experiments

	Chloroform	Acrylonitrile	(* experimental values)
Aqueous Phase	x _{Water} = 0.9996	x _{Water} = 0.947	x _{Water} = 0.946
	R _{recovery} = 97.8%	R _{recovery} = 92.6%	R _{recovery} = 89%
Entrainer Phase	x _E = 0.9990	x _E = 0.712	x _E = 0.6916
	R _{recovery} = 81%	R _{recovery} = 72%	R _{recovery} = 69%
Final Still	x _{Chloroform} = 0.9945	x _{Chloroform} = 0.992	x _{Chloroform} = 0.995
	R _{recovery} = 99.2%	R _{recovery} = 95.6%	R _{recovery} = 91.8%

CONCLUSIONS

- Pressure swing distillation is a privileged option. It doesn't involve additional entrainer. Simple performance in one or two batch distillation column operating at different pressures.
- Heterogeneous entrainers have a more privileged position than homogeneous. Higher number of ternary diagram match with feasible rules
- Heterogeneous entrainers in HABD: little amount of entrainer, separatrix can be crossed by the still path, simplest batch distillation sequence, more flexible reflux policy (entrainer-rich phase or both decanted phases)
- Heterogeneous entrainers in HEBD: Complex ternary residue curves are feasible, withdrawal of saddle binary heteroazeotrope at the column top by feeding E at the top of the column, more flexible reflux policy as HABD
- Water seems a promising candidate for treatment organic wastes by HEBD and HABD. cheap, non toxic, non pollutant