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# Non-conventional heterogeneous batch distillation process for separating an organic waste mixture

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**Objectives:** Separation of the chloroform – methanol mixture by Heterogeneous Extractive Batch Distillation (HEBD). Feasibility studies, rigorous simulation and experimental validation.

**Chloroform (A) + Methanol (B):** An organic waste at it is an effective solvent for the extraction of bioactive substances from biological sources. Minimum boiling Azeotrope: T = 53.5 °C, X<sub>A</sub>= 0.65

## 1 - Selecting Entrainer with RegSolExpert®

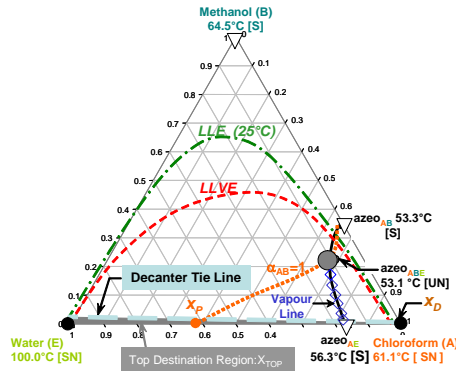
### Heterogeneous entrainer versus Homogeneous Entrainer

- Entrainer can form azeotropes with A and B
- Entrainer must form a saddle binary heteroazeotrope with A or B
- Univolatility line  $\alpha_{AB}=1$  must intercept the binary heterogeneous side AE or BE
- Entrainer fed at the column top. Extractive column section ONLY exist
- Brings MORE design alternatives because offers MORE suitable entrainers

Entrainer (E)	T <sub>Boiling</sub> (°C)	$\gamma_A^s/\gamma_B^s$	process	x <sub>p</sub>	Rejection
chloroform (A)	61.1	0.19			
methanol (B)	64.5				
Acetic acid	118.0	1.87	BED	0.42 (A)	1,3
Allyl alcohol	96.9	1.23	BED	0.54 (A)	1, 2, 3
1-Propanol	97.4	1.50	BED	0.43 (A)	3
1-Butanol	117.8	1.32	BED	0.22 (A)	3
2-Methyl-1-Propanol	107.6	1.32	BED	0.29 (A)	3
2-butanol	99.5	1.24	BED	0.24 (A)	3
Water	100.0	398.22	HBED	0.42 (A)	-
n-Butyl acetate	126.5	0.11	BED	0.66 (B)	3
Furfural	161.4	0.42	BED	0.60 (B)	1, 2, 3
Pyridine	115.2	0.52	BED	0.46 (B)	2, 3
Nitrobenzene	210.6	0.05	BED	0.70 (B)	1, 2, 3
4-methyl-2-pentanone	116.7	0.18	BED	0.61 (B)	2, 3
1,4 dioxane	101.3	0.13	BED	0.73 (B)	1, 2, 3
Methyl cyclohexane	101.0	0.02	HBED	0.55 (B)	2, 3

Rejection criteria: <sup>1</sup> toxic, <sup>2</sup> pollution, <sup>3</sup> cost

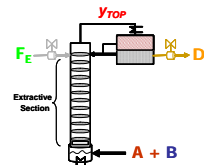
## 2 - Residue Curve Map



### Water is the best entrainer

- Huge improvement of  $\gamma_A^s/\gamma_B^s$  ratio : excellent separation efficiency
- x<sub>p</sub> value > 0.4 guarantee a moderate entrainer consumption
- cheap, non toxic, non pollutant
- The process is non conventional: heterogeneous batch extractive distillation

## 3 – Column configuration



### Column Features:

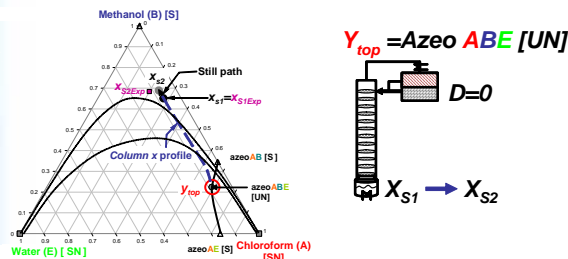
Initial Charge=20 mol  
Theoretical trays=45  
P=1 atm, T<sub>decanter</sub>=25°C  
Heat Duty= 150 W  
Liquid Holdup:  
Tray=0.0015L  
Decanter=0.05L  
Total Condenser=0.02L

## 4 – HEBD process in 5 steps + Simulation with ProsimBatch® + Experiments (for steps 1,2,3 and 4)

### Column Start Up:

**Step 1 - Heat Up (12 min):** Heat the still (x<sub>s1</sub>) and fill up the column.

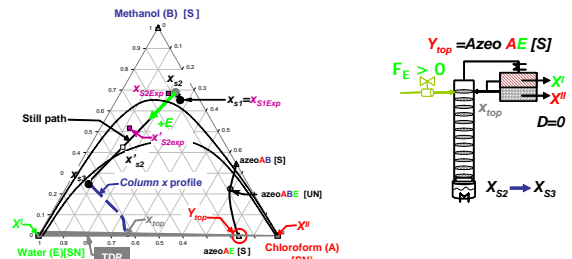
**Step 2 - R∞:** Still Composition x<sub>s2</sub> and Y<sub>top</sub>=Azeo ABE. t=120 min



### Extractive Distillation: F<sub>E</sub>=20 mol/h

**Step 3 - R∞ (D=0):** x<sub>s2</sub> moves towards E, Y<sub>top</sub>=Azeo AE [S]

t=100 min Liquid profile finishes at x<sub>top</sub>  
Decanter two liquid phases: X' and X''

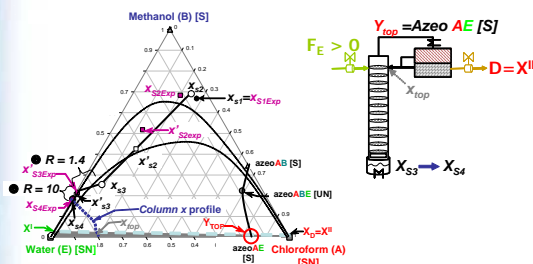


### Extractive Distillation: F<sub>E</sub>=20 mol/h, D>0

**Step 4 - R finite:** Withdrawal of Chloroform X<sub>D</sub>=X'' (heavy phase)

R=1.4 t=60 min x<sub>s3</sub> influenced by +E and -D

R=10 t=20 min Still path finishes at Binary Side BE

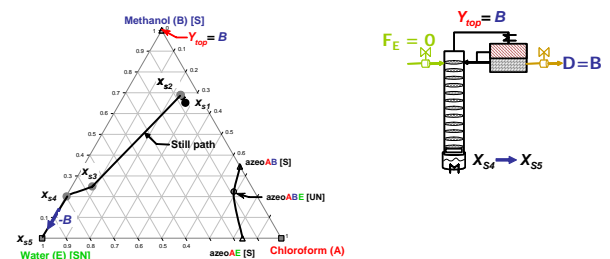


### Conventional Distillation: Separation of BE (Simulation Results only)

**Step 5 - R finite F<sub>E</sub> = 0:** Withdrawal of methanol X<sub>D</sub>=B

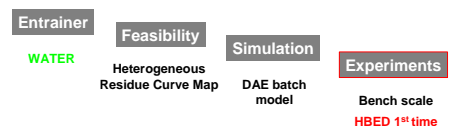
R=1.5 t=100 min Final still composition x<sub>s5</sub>=E

R=10 t=150 min



### CONCLUSIONS

- Validation of the Heterogeneous batch extractive distillation process



- Good chloroform and methanol purity and recovery

	SIMULATION		EXPERIMENTS	
	Amount (mol)	Purity	Amount (mol)	Purity
Aqueous Phase Decanter	0.045	x <sub>E</sub> 0.9957	1.07	x <sub>E</sub> 0.9690
Chloroform Distillate	5.2	x <sub>A</sub> 0.9906	4.86	x <sub>A</sub> 0.9914
Chloroform Recovery		95.4 %		89.1 %
Final Still (Step 4)	72.3	x <sub>A</sub> 0.0001 x <sub>B</sub> 0.1795 x <sub>E</sub> 0.8204	69.2	x <sub>A</sub> 0.0000 x <sub>B</sub> 0.1664 x <sub>E</sub> 0.8336
Methanol Recovery		96.6 %		85.8 %