Cyclic Separation:
New trays and Experimental Results

EFCE WP Fluid Separations
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Abildskov J., Bay Jørgensen S.
CAPEC-PROCESS
Department of Chemical and Biochemical Engineering
Technical University of Denmark

Outline

Cycled Trays Operation
- Theoretical Performance: Lewis Case II analogy
- Early realizations; Simultaneous Draining
- Simultaneous Draining; Problems
- Ideas to approach theoretical performance

New Principles of Design and Operation

New Trays and Sequential Tray Draining

New Realization

Results

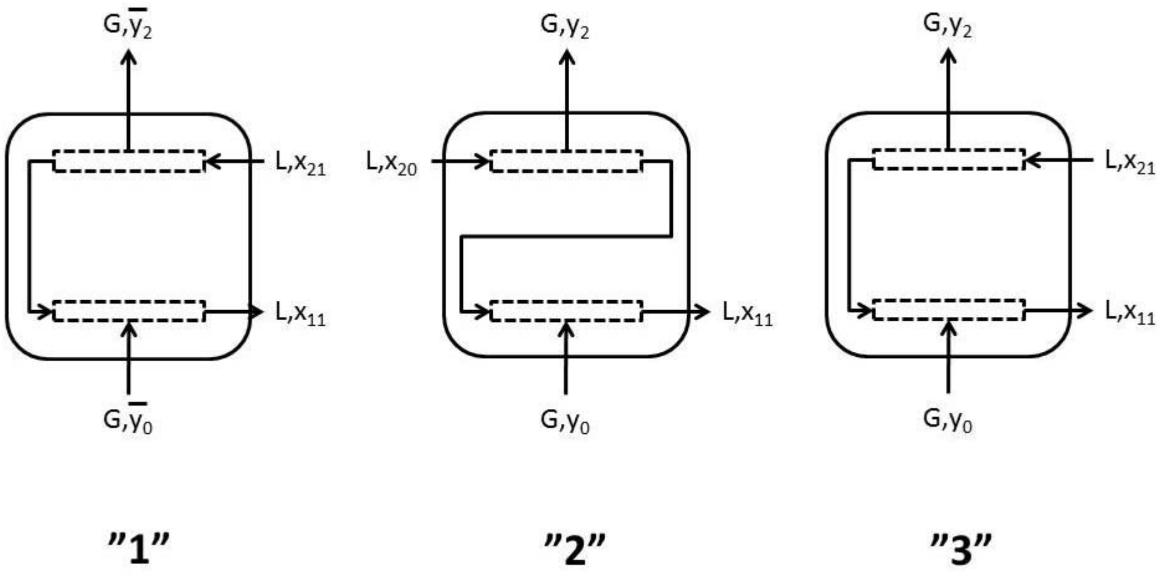
Future Directions

Conclusions

Other Explorations

- AIChE Spring Meeting 2016
- C. Knoesche, J. Paschold, S. Buetehorn (BASF):
- Paper 87b: “Cyclic Operation of Distillation Columns”
- Maleta CD

Theoretical Performance: Lewis Case II Analogy



Theoretical Performance: Lewis Case II Analogy

CASE	"I"	"II"	"III"
$\varepsilon_{tray}/\varepsilon_{point}$	1.7	2	1.5

$\varepsilon_{point} \rightarrow 1$

- Performance:

  "II" > "I" > "III"

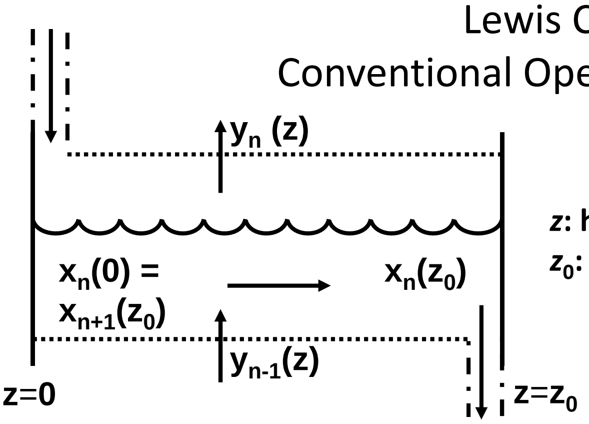
- (Convenience of) realization:

  "III" > "I" > "II"

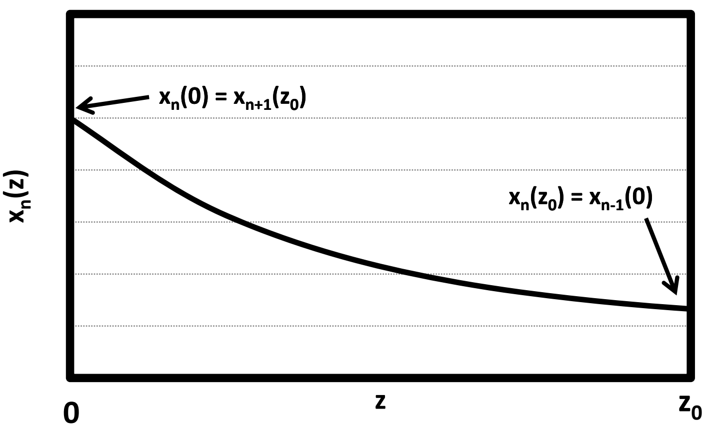
" … (Lewis' case III) …. is probably the most common in conventional operation because of designers' understandable reluctance to install downcomers in the configuration of case II" (Sommerfeld et al., 1966)

Theoretical Performance:

Lewis Case II Analogy; Conventional Operation with Downcomers



z: horizontal tray position
z_0: length of liquid flow



→ Liquid in down-comer from n+1 enters plate n at z = 0: $x_n(0) = x_{n+1}(z_0)$

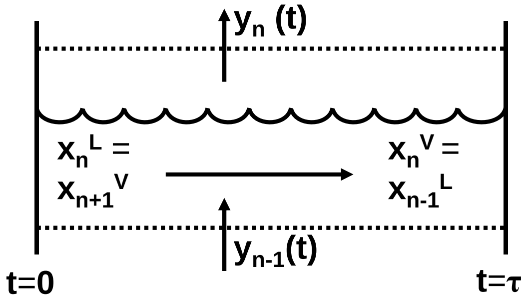
→ No liquid back-mixing; progressive depletion of volatile component by contact with vapor from plate n – 1

→ $z = z_0$: $x_n(z_0)$ is discharged to n – 1

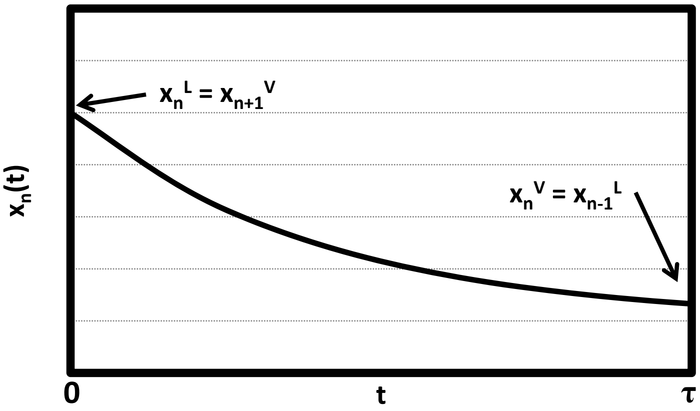
Theoretical Performance:

Lewis Case II Analogy;

Cyclic Operation without downcomers



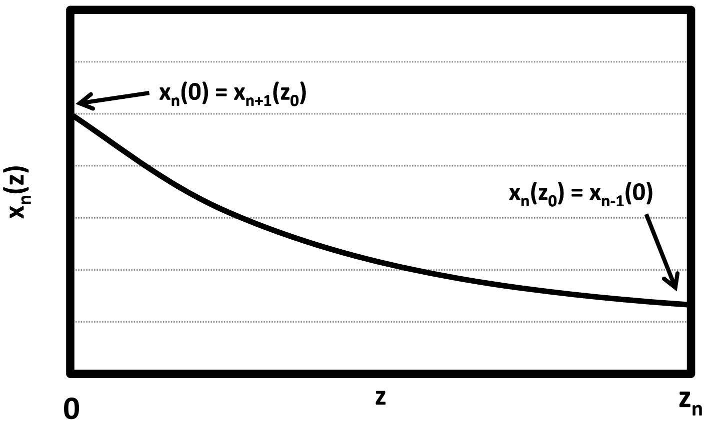
Independent variable: t
Time length: VFP = τ



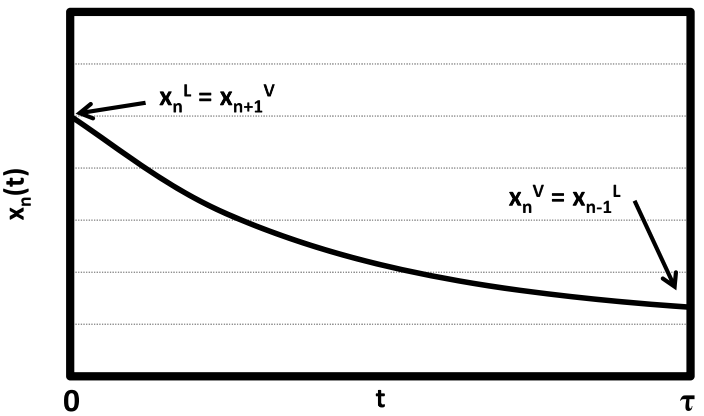
- → No liquid phase enrichment on plate *n* by liquid flowing down
- → No liquid back-mixing in time domain
- → Depletion of volatile component occurs monotonically with time
- → Perfectly mixed plate
- → Amount of material dropped during LFP is exactly equal to one plate holdup

Theoretical Performance: Lewis Case II analogy

- Sommerfeld (1966)



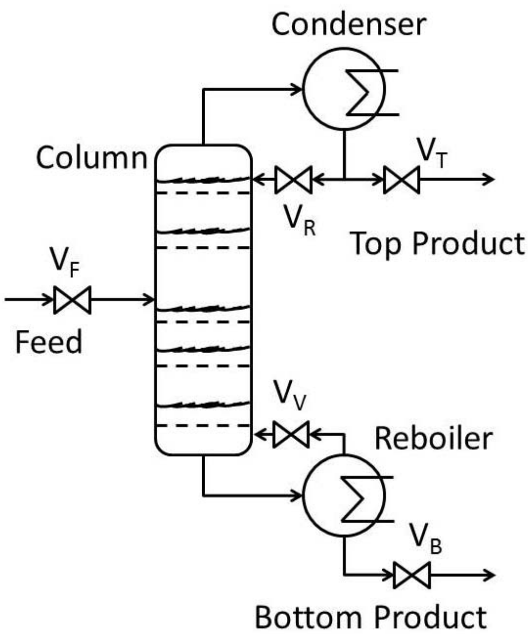
z: horizontal tray position



t: time of vapor flow period of duration τ

Early realizations; *Simultaneous Draining*

- Three 1961 papers (Cannon)
- *Exactly one* plate holdup must be dropped during LFP
- Basis of the generation: *Simultaneous Draining*
- TP = VFP + LFP
- Modeling:
  - ❑ McWhirter (1963)
  - ❑ Sommerfeld et al. (1966)
  - ❑ Chien et al. (1966)
  - ❑ Robinson/Engel (1967)



Vapor Flow Period (VFP):
V_F, V_R, V_T and V_B are closed. V_V is open.

Liquid Flow Period (LFP):
V_T, V_B, V_F, V_R are opened. V_V is closed.

Simultaneous Draining; Problems

- Back-Mixing
- Non-uniform draining of trays
  - ❑Schrodt V. N. et al., 1967. Chem. Eng. Sci., 22, 759
  - ❑Gerster J.A., Scull H.M., 1970. AIChE J. , 16, 108.
  - ❑Larsen J., Kümmel M., 1979. Chem. Eng. Sci., 34, 455.
- Pressure Dynamics
- Liquid oscillations on trays

Approaches to Theoretical Performance - Improved Liquid Flow Control

- Matsubara
  - Collect liquid in a store, prior to transfer to the tray below.
  - Vapor transferred in tubes from tray to tray
- Furzer (TD) Time delay trays
  - Gutter channels to delay the liquid draining
  - Baffles to avoid oscillations
- Hentrich/Vogelpohl (1980)
  - 1st approach: Liquid was led into a storage tube; when all trays were empty, the liquid was released into the next tray. This was accomplished using a central rod in the column (EXPENSIVE).
  - 2nd approach: Sieve tray with packing material below, to make the liquid drain more slowly during the LFP
- Baron: Stepwise Periodic Operations
- Maleta CD (21st century): Sluice chambers

New Principles of Design/Operation

Tray Design Principle:

The periodic cycled separation tray should be constructed such that:

1) All the liquid leaves the tray and enters the tray below *without being mixed* with any other liquid during the liquid drainage
2) *Vapor flow can continue* during liquid drainage

Operational Principle for Sequential Tray Draining:

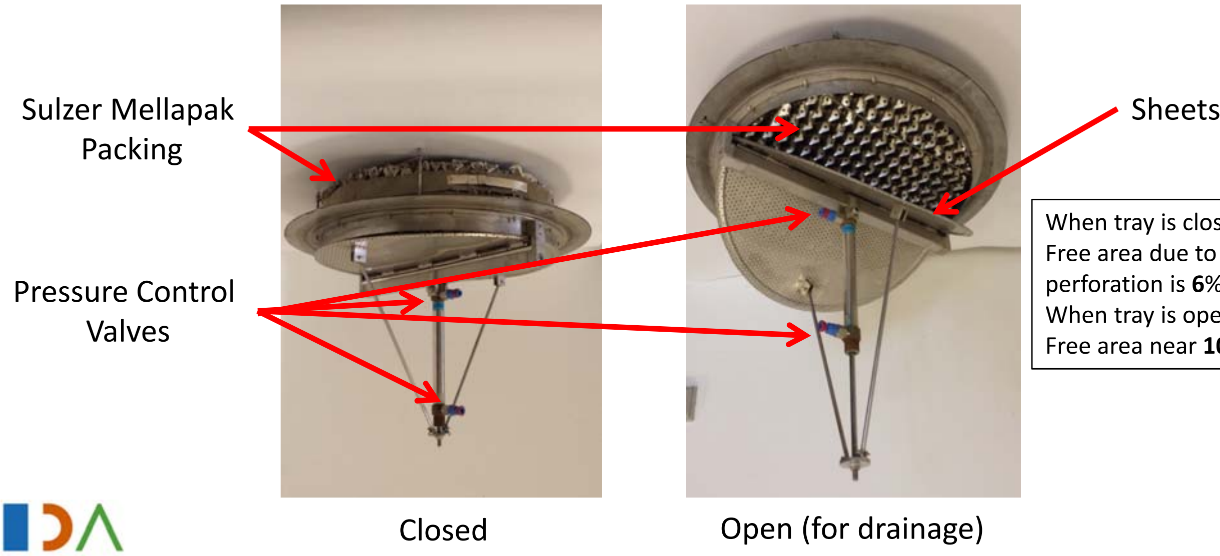
To minimize mixing with liquid from other trays enforce a special operational principle for Sequential Tray Draining:

The trays are emptied *sequentially rather than simultaneously*, while the vapor flow is maintained throughout

New Realization; Trays

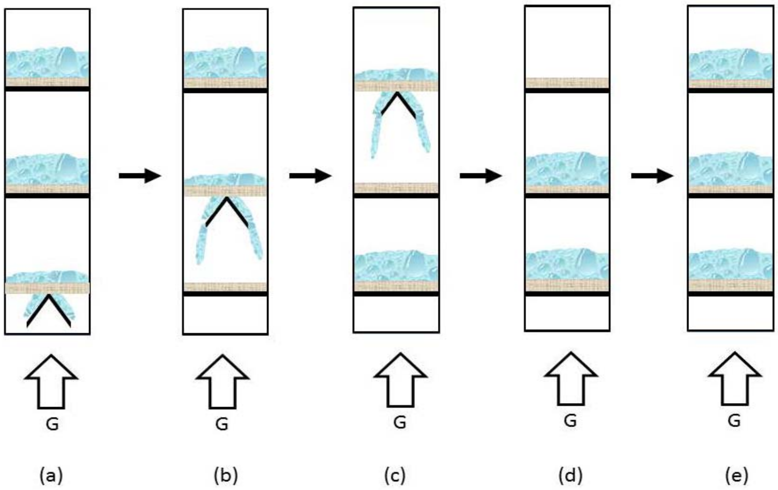
Cyclically Operated Perforated Sheets (COPS)

Layer of structured packing for liquid oscillation damping

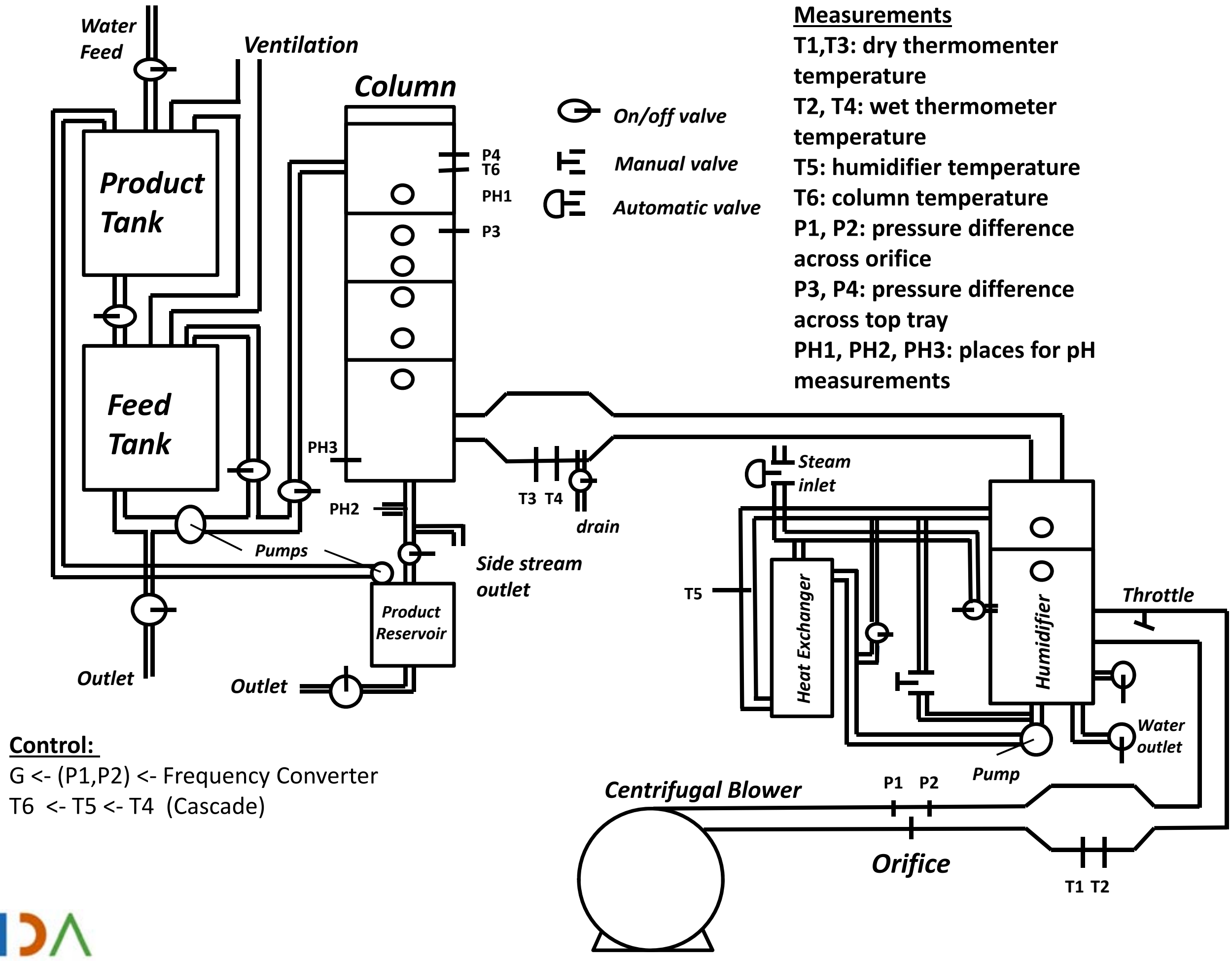


When tray is closed:
Free area due to perforation is **6%**
When tray is open:
Free area near **100%**

New Realization; Sequential Tray Draining



Sequential tray drainage operation for a column with three trays. When the top tray is empty (d) a load of liquid is fed to the top tray (e). The column remains in state (e) for the remainder (if any) of the cycling period time until a new liquid drainage sequence is initiated at the start of the next period (a) – (c).



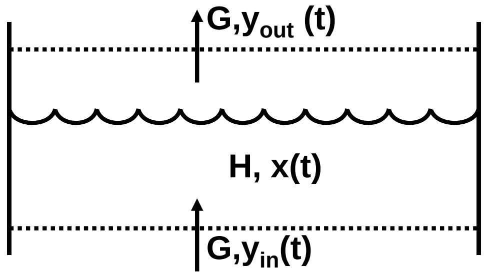
Control:

G <- (P1,P2) <- Frequency Converter

T6 <- T5 <- T4 (Cascade)

Results; Point Efficiency, ε_t

- Strip NH_3 from water (Gerster/Scull, 1970)
- Tray molar balance + Murphree expression:
  → Slope of pH versus t gives ε_t



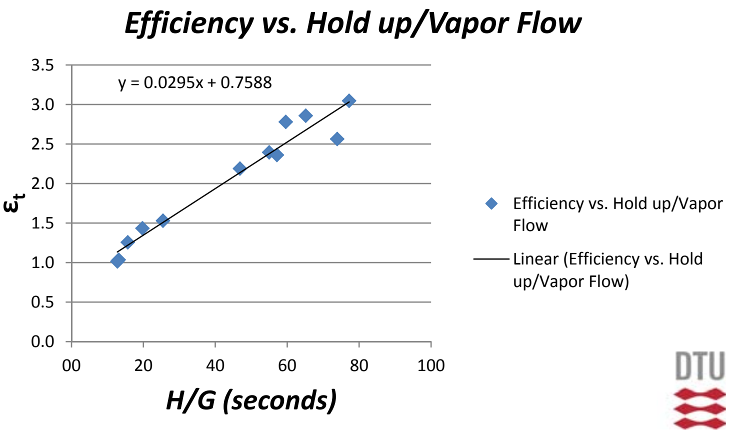
$$H\frac{dx}{dt} = G(y_{in} - y_{out}) \quad \Rightarrow$$

$$\frac{dx}{dt} = -\varepsilon_t \frac{G}{H} m \cdot x \quad \Rightarrow \quad \frac{dx}{dt} + \varepsilon_t \frac{G}{H} m \cdot x = 0$$

$$\Rightarrow \quad \varepsilon_t = \frac{H}{Gmt}\left[\log c(t=0) - \log c(t)\right]\ln 10$$

$$= \frac{2H}{Gmt}\left[pH(t=0) - pH(t)\right]\ln 10$$

- Assume: Only NH_3 transfer
- CO_2 Controversy



Results; Column Efficiency, Experimental Procedure

Set (L,G) → $FP = \frac{L}{G}\sqrt{\frac{\rho_G}{\rho_L}}$ → Obtain Stationary Periodic State → Measure (x_f, x_p) by titration → $S = \frac{x_f(1-x_p)}{x_p(1-x_f)}$

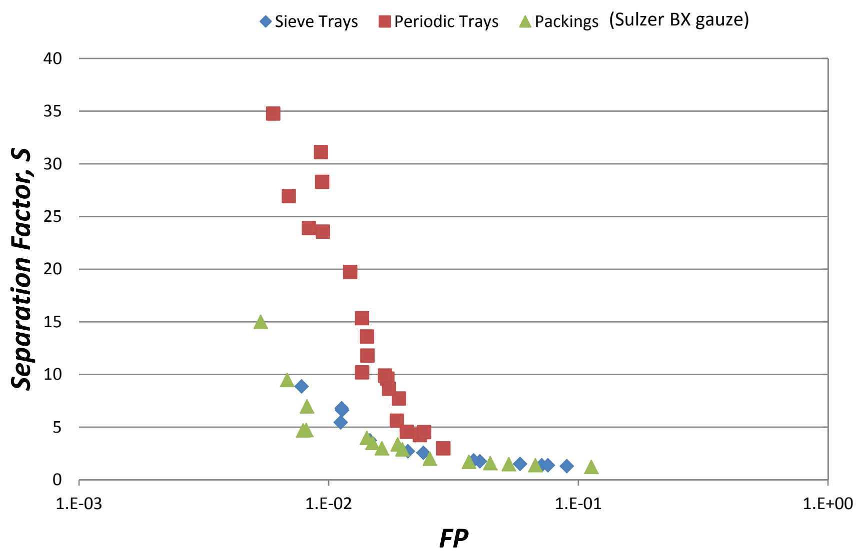
↓

$m = 0.073 \times 10^{6.695-\frac{1665}{T}}$ (Gerster,1958)

→ $N_{id} = \frac{\ln\left[(1-\frac{1}{\lambda})\frac{x_f}{x_p} + \frac{1}{\lambda}\right]}{\ln\lambda}$, $\lambda = \frac{mG}{L}$

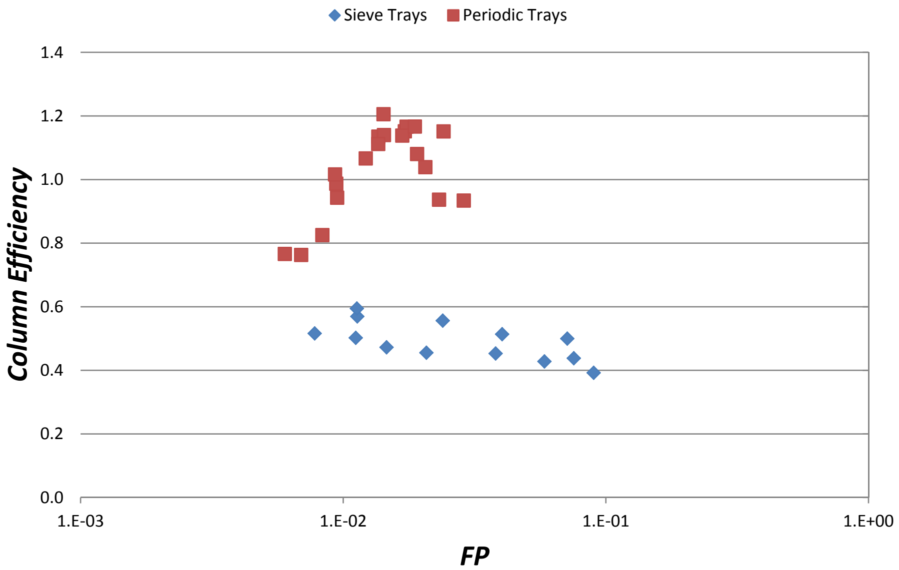
→ $CE_p = \frac{N_{id}}{3}$, $CE_c = \frac{N_{id}}{3}$

Results; Separation Factors

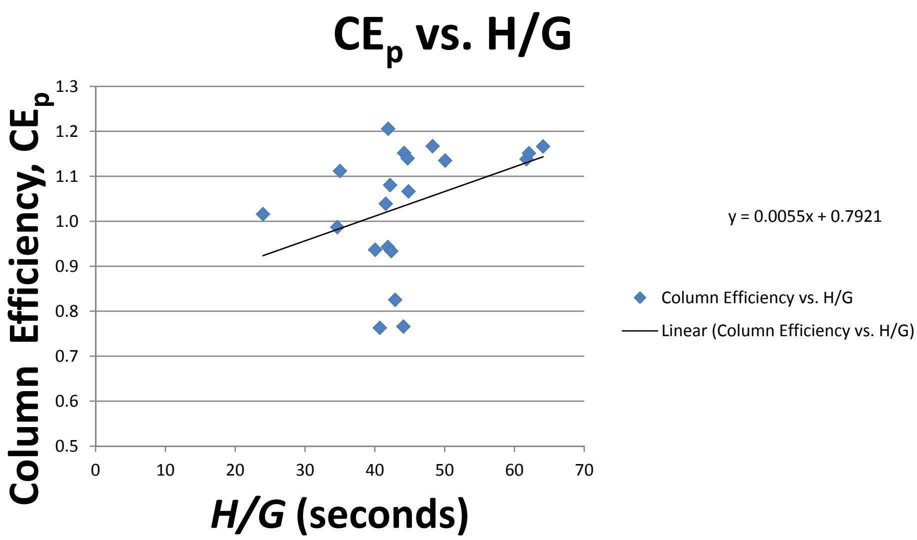


Stream	Limits
G (mol/s)	5-10
L (mol/s)	0-20

Results; Column Efficiencies



Results; Influence on Tray Efficiency of Flows

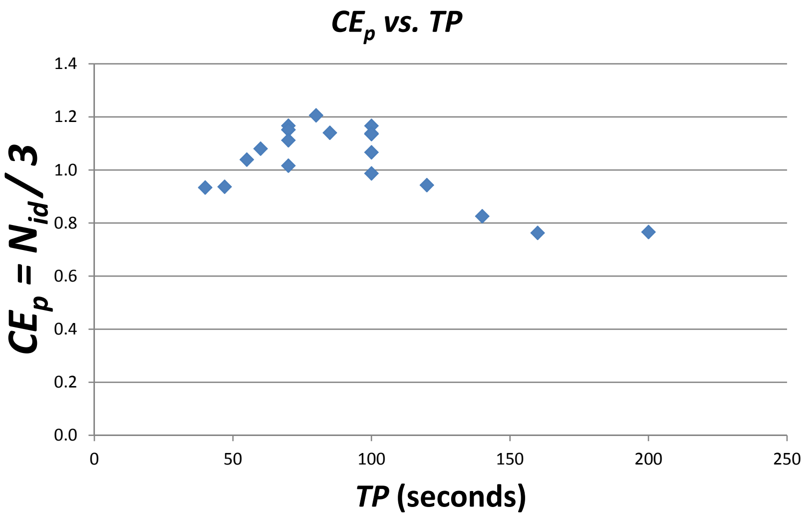


No points at H/G > 70 …….!

Results; Influence on Column Efficiency of Period Lengths

- Maximum?

TP: Total Period Duration



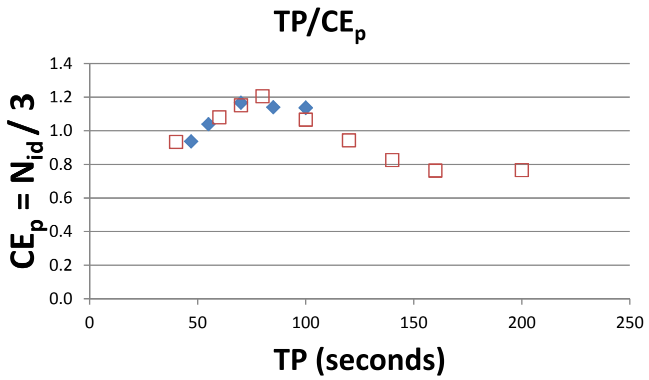
$$N_{id} = \frac{\ln\left[\left(1-\frac{1}{\lambda}\right)\frac{x_f}{x_p}+\frac{1}{\lambda}\right]}{\ln(\lambda)}$$

$\lambda = mG/L$

Experimental data make N_{id} go thru extremum

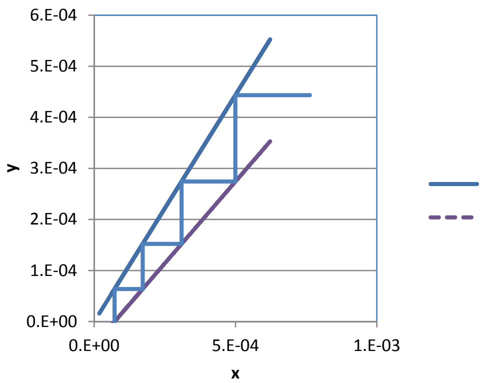
$6.9 < G(mol/s) < 8.4$
$2 < L (mol/s) < 8.8$

Why optimum TP?



Blue:
G = 7
dP = 50

Red:
G = 8.3
dP = 60



Equilibrium Curve:
$y^* = x \cdot m$
Operating Line:
$y = (x-x_p)\cdot(L/G)$

TP↑ ~ L↓~ (L/G)↓ ~ N_{id} ↓

BUT if

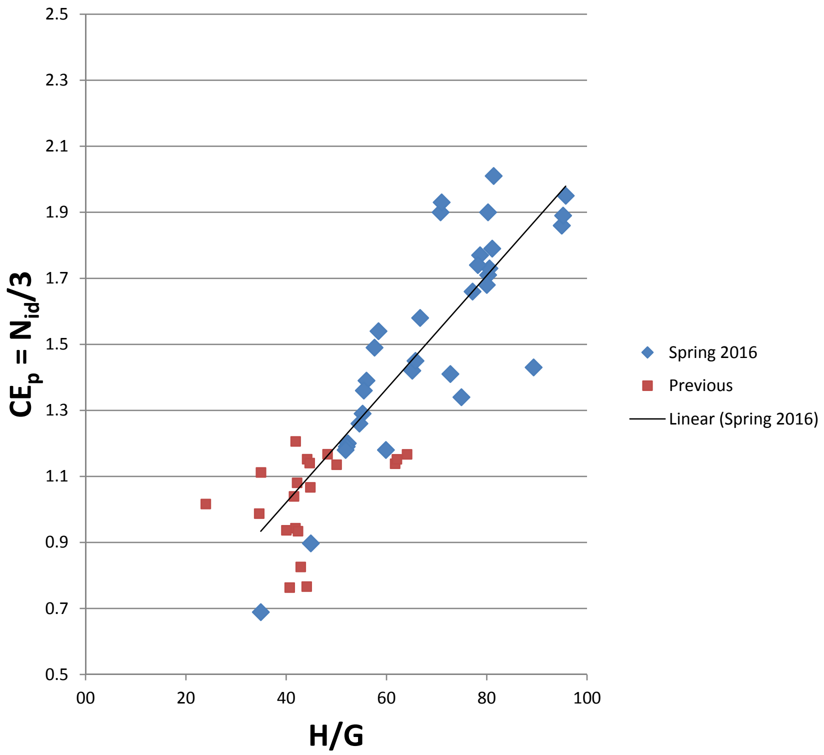
x_p ↓ N_{id} ↑

This may compensate, if x_p gets sufficiently low. This seems to be the case at low TP values, but only up to 80 seconds.

Further Work

- Operation at H/G beyond 70 seconds?

Latest Results



Conclusions

- Presented:
  - New Tray Design (Principle): COPS
  - New Operational Principle: Sequential Draining
  - Steady Operation: Avoiding Pressure Dynamics and Liquid (Back) Mixing
- Future Directions:
  - Operation at higher (periodic) tray efficiencies