

### **Distillation modelling and control**

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# Sigurd Skogestad

- 1955: Born in Norway
- 1978: MS (Siv.ing.) in chemical engineering at NTNU
- 1979-1983: Worked at Norsk Hydro co. (distillation)
- 1987: PhD from Caltech (supervisor: Manfred Morari)
  - Thesis: "Studies om robust control of distillation columns"
- 1987-present: Professor of chemical engineering at NTNU
  - 42 Phd students graduated (9 with distillation in title of thesis)
- 1999-2009: Head of Department
- 2015- : Director SUBPRO
- 200+ journal publications
- Book: Multivariable Feedback Control (Wiley 1996; 2005)
- Book: Chemical and energy process engineering (CRC Press, 2008)
  - 1989: Ted Peterson Best Paper Award by the CAST division of AIChE
  - 1990: George S. Axelby Outstanding Paper Award by the Control System Society of IEEE
  - 1992: O. Hugo Schuck Best Paper Award by the American Automatic Control Council
  - 2006: Best paper award in Computers and chemical engineering.
  - 2011: Process Automation Hall of Fame (US)
  - 2012: Fellow of American Institute of Chemical Engineers (AIChE)
  - 2014: Fellow of International Federation of Automatic Control (IFAC)
  - 2019: Best paper award at the ESCAPE 2019 Symposium (Eindhoven)
  - 2019: Computing in chemical engineering award from the American Institute of Chemical Engineers











#### Geiranger fjord





# Outline

- 1. Introduction / Distillation as a separation process
- 2. Modelling
- 3. Dynamics
- 4. Optimal operation
- 5. Control



# 1. Basis: Difference in boiling points (volatility)



Relative volatility:  $\alpha = \frac{K_L}{K_H} = \frac{y_L/x_L}{y_H/x_H}$   $\ln \alpha \approx \underbrace{\frac{\Delta H^{vap}}{RT_B}}_{\text{typical:}\approx 9-15} \cdot \underbrace{\frac{\Delta T_B}{T_B}}$ 

 $\Delta H^{vap}$  - avg. heat of vap. at  $T_B$  [kJ/mol]

 $\Delta T_B$  - boiling point difference [K]  $T_B$  - geometric avg. boiling point [K] L – light component H – heavy component

Example. iso-pentane (L) – pentane (H). Boiling points: 28 °C(L) and 36.2 °C(H)  $\Delta T_B = 8.2 \text{ K}, \quad \alpha \approx e^{10 \cdot \frac{8.2}{300}} = 1.32$ 

I.J. Halvorsen and S. Skogestad, ``Distillation Theory'', In: Encyclopedia of Separation Science. Ian D. Wilson, Academic Press, 2000, pp. 1117-1134.



### Batch distillation with no reflux (-> N=1)



Abb. 11. Destillationsapparatur mit Kühlung in zwei Rohren, nach [44].

1545





Abb. 13. Destillationsapparatur mit Kühlung in vertikaler Kühlschlange, nach [46].

1593

2020



### **Continuous distillation with reflux (L)**





# When use distillation?

- Liquid mixtures with difference in boiling point
- Unbeatable for high-purity separations •
  - Essentially same energy usage independent of purity!

 $\frac{V_{min}}{F} \approx \frac{1}{\alpha - 1} + \frac{D}{F}$ 

Number of stages increases only as log of and many stages ٠ Fenske:  $N_{min} = \ln S / \ln \alpha$ 

- Close-boiling mixture ( $\alpha$  close to 1)
  - Need a lot of energy (heat)

- Well suited for scale-up
  - Columns with diameters over 15 m **》**
- Examples of unlikely uses of distillation:
  - High-purity silicon for computers (via SiCl<sub>3</sub> distillation) **》**
  - Water heavy-water separation (boiling point difference only **》** 1.4°C)



### «Distillation is an inefficient process»

- This is a myth!
- By itself, distillation is an efficient process
  - Typically, thermodynamic efficiency >50%
- It's the heat integration that may be inefficient.
  - Yes, it can use a lot of energy (Q<sub>r</sub>=heat), but it provides ~ the same energy as cooling (Q<sub>c</sub>) at a lower temperature





### Thermodynamic efficiency of distillation



$$\eta = \frac{W_s^{id}}{W_{s,carnot}} = \frac{-FRT_0 \sum_{i=1}^{N} z_i \ln z_i}{Q_r T_0 (\frac{1}{T_c} - \frac{1}{T_H})}$$

Separation into pure components

 Plot is for liquid feed, binary mixture

$$\eta = \frac{W_s^{id}}{W_{s,tot}} = \frac{-(z \ln z + (1-z)\ln(1-z))}{(z+\frac{1}{\alpha-1})\ln\alpha}$$

Plot from I.J. Halvorsen.

Ref: S. Skogestad, Chemical and Energy process engineering, CRC Press, 2009, pp. 224 Ref: C.J. King, Separation processes, McGraw-Hill, 1971, 1980



 $\alpha$  = relative volatility

### 2. Simple to model. Equilibrium stage concept

- (i) Component material balances  $\frac{dN_{ij}}{dt} = L_{i+1}x_{i+1,j} + V_{i-1}y_{i-1,j} - L_i x_{i,j} - V_i y_{i,j}$
- (ii) Overall material balance (flow dynamics)

$$\frac{d}{dt}M_i = \frac{d}{dt}(M_{iL} + M_{iV}) = L_{i+1} + V_{i-1} - L_i - V_i$$

(iii) Energy balance

$$\frac{dU_i}{dt} = L_{i+1}h_{L,i+1} + V_{i-1}h_{V,i-1} - L_ih_{Li} - V_ih_{Vi} \quad \text{where} \quad U_i = M_{Li}$$

(iv) Algebraic relations for hydraulics and pressure drop

$$L_i = f_1(M_{Li}, V_i, \Delta p_i); \quad V_i = f_2(M_{Li}, \Delta p_i)$$

(v) Algebraic equations for VLE between phases:  $y_i = K(x_i, p, T)$  etc..

VLE = Vapor-Liquid Equibrium



 $u_{Li} + M_{Vi} u_{Li}$ 

Francis weir formula:  $L_i = M_{L_i}^{2/3}$ 

VLE: "Relative volatility model" Usually most important!

- Activity coefficient (e.g. UNIFAC)
- Or: Equation of state (e.g. SRK, PR)



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$$\frac{d}{dt}M_i = \frac{d}{dt}(M_{iL} + M_{iV}) = L_{i+1} + V_{i-1} - L_i - V_i$$



#### Modelling. Adjusting parameters

- 1. VLE. May need experimental data
- 2. Stages (N)
  - Use steady-state model
  - Match steady-state compositions and temperature profiles
    - Adjust N to get theoretical stages in each section (also for packed column)
    - Tray column: Can use Murphee efficiency. I use bypass on V and/or L
- 3. Holdup Mi. Match with dynamic data for compositions
  - Typical column holdup, M<sub>L</sub> = ΣM<sub>i</sub> Tray column: 10% of column volume Packed column: 5% of column volume
- 4. Liquid dynamics: Make step in L and see how long it takes for change to reach bottom
  - Francis weir formula:  $\theta_L \approx (2/3) M_L / L$



# 3. Dynamics

Example . Propylene-propane (C3-splitter, column D)

- **4** N = 110 theoretical stages
- $\Delta T_{B} = 5.6 K$
- $\alpha = 1.12$  (relative volatility at 15 bar)
- **4** Purities: 99.5% propylene (top) and 90% propane (bottom)
- **4** Assume constant molar flows

$$L/D = 19, D/F = 0.614$$

$$\frac{4}{F} \quad \frac{M_{tot}}{F} = \frac{\sum_{i} M_{i}}{F} = 111 \text{min}$$



#### External flow change Propylene-propane. Simulated composition response. Increase reflux 0.4% ( $\Delta L = +0.05$ , $\Delta D = -0.05$ ) with V constant:





#### Increase in L +0.05





#### External flow change. Propylene-propane. Simulated composition response with detailed model. Increase boilup (V: +0.05, D: +0.05) with L constant





- What happens if we increase both L and V at the same time?
- Then D and B are constant
- Internal flow change





Internal flow change.

Propylene-propane. Simulated composition response with detailed model. Increase both L and V by same amount ( $\Delta V = \Delta L = +0.05$ )





## **External and Internal Flows**



 $\Delta L = \Delta B = -\Delta D = (-0.1, -0.01, 0, 0.01, 0.1)$ 

Large effect on composition (large "gain") One products get purer – the other less pure Internal flows (10X)



 $\Delta$  V= $\Delta$  L = (1, 0, -1).  $\Delta$  B= $\Delta$  D=0

Small effect on composition (small "gain") Both products get purer or both less pure

«ILL-CONDITIONED INTERACTIVE PROCESS». Problem for control?



# Liquid flow dynamics are essential for control



- Liquid flow dynamics "break" the initial two-way interaction between top and bottom.
  - Want to close one loop (temperature or composition) with closed-loop time constant faster than the liquid flow dynamics

S. Skogestad, "Dynamics and control of distillation columns - A tutorial introduction", Trans IChemE, Part A (Chemical Engineering Research and Design), 75, Sept. 1997, 539-562



Condenser

# Nonlinearity

Use logarithmic compositions

$$X_i = ln \frac{x_{Li}}{x_{Hi}}$$

$$X_i \gg ln \frac{T_{btm} - T_i}{T_i - T_{top}}$$

S. Skogestad, ``Dynamics and Control of Distillation Columns - A Critical Survey", *IFAC-symposium DYCORD+'92*, Maryland, Apr. 27-29, 1992. Reprinted in *Modeling, Identification and Control*, Vol. 18, 177-217, 1997.





Figure 7: Nonlinear response in distillate composition for changes in L of 0.1%, 1%, 10% and 50%. Right plot: Logarithmic composition

S. Skogestad, "Dynamics and control of distillation columns - A tutorial introduction", Trans IChemE, Part A (Chemical Engineering Research and Design), 75, Sept. 1997, 539-562



# **Conclusion dynamics**

- Dominant first order response often close to integrating from a control point of view
- Liquid flow dynamics decouples the top and bottom on a short time scale, and make control easier
- Logarithmic transformations linearize the response



### 4. Optimal operation distillation column



- Steady state (control levels and pressure): 2 degrees of freedom (e.g. L,V)
- Optimal operation: Minimize cost J subject to satisfying constraints
- Cost to be minimized (economics)

$$J = -P \quad \text{where} \quad P = p_D D + p_B B - p_F F - p_V V$$

$$value \text{ products} \quad cost \text{ feed}$$

$$F_{,z_F}$$

$$K_{D, \text{ impurity}} < max$$

$$K_{B, \text{ impurity}} < max$$

Column capacity (flooding):  $V < V_{max}$ , etc.



### **Expected active constraints distillation**

#### • Valueable product: Purity spec. always active

- Avoid product "give-away" ("Sell water as methanol")
- Saves energy

#### Control implications:

- 1. Control valueable product at spec.
  - Control D at 0.5% water

#### 2. Overpurify other end to reduce loss

- Control B with methanol < 2%
- Overpurifying may not cost much energy (V) if enough stages
- If "few" stages: May be optimal to operate at max energy (V) to minimize loss of valuable product



water

+ max. 2% methanol



# 5. Control

#### DISTILLATION CONTROL

 Studied in hundreds of research and industrial papers over the last 40 years



Problem industrial papers: Ad-hoc Problem academic papers: Not distillation, but show that "my control theory is best"

S. Skogestad, <u>"The dos and don'ts of distillation columns control"</u>, Chemical Engineering Research and Design (Trans IChemE, Part A), **85** (A1), 13-23 (2007).



# Two objectives for control

Have 5 dynamic degrees of freedom

### 1. Stabilize (avoid drift)

- Levels (M<sub>D</sub>, M<sub>B</sub>, pressure)
- Temperature profile («level of heavy component»)

NOTE: Temperature setpoint can be used as degree of freedom for composition control

#### 2. Optimize operations

Normally control product compositions (x<sub>D</sub>, x<sub>B</sub>)



# **Issues distillation control**

- The "configuration" problem (pairings for level and pressure)
  - Which are the two remaining degrees of freedom?
    - e.g. LV-, DV-, DB- and L/D V/Bconfigurations
- The temperature control problem
  - Which temperature (if any) should be controlled?
- Composition control problem
  - Control two, one or no compositions?





# Stabilize temperature profile ("level of heavy component")



- Temperature sensor should be located at «sensitive» stage (with high gain)
- Typically in middle of top or bottom section



# Which stage? Binary column



slope closely correlated with steady state gain



## **Multicomponent column**



Conclusion: Temperature slope alone OK only for binary columns



### Which configuration? Level control top: Use L or D?

Would like to use D because L is most effective for composition control



However, for large L/D (>5): Seems almost impossible to control level with D (small range because need D>0)

#### FORTUNATELY:

- 1. Tight level control is NOT important
- 2. Fast inner temperature loop gives indirect level control!

Conclusion: Recommend using D (and B) for level control!! Gives LV-configuration



# **Conclusion configurations**

- Normally use LV-configuration
   because it is
  - simplest
  - level tunings do not matter for column behavior
  - can get smooth variations in product rates D and B
- Usually add temperature loop using L or V
  - Especially for difficult separations with large L and V
  - Gives indirect level control
  - Breaks interactions





# LV-configuration with temperature loop and dual composition control



- Inner fast T-loop should be in the «important» end
  - The figure above is when bottom composition is most important
- Temperature sensor should be located at «sensitive» stage.



# Myth of slow control

• Let us get rid of it!!!

Compare manual ("perfect operator") and automatic control for column A:

- 40 stages,
- Binary mixture with 99% purity both ends,
- relative volatility = 1.5
- L/D = 5.4
- First "one-point" control: Control of top composition only
- Then "two-point" control: Control of both compositions

S. Skogestad, "Dynamics and control of distillation columns - A tutorial introduction", Trans IChemE, Part A (Chemical Engineering Research and Design), 75, Sept. 1997, 539-562



### Myth about slow control One-point control



Figure 12: One-point control of  $x_D$ : Response to a 1% step increase (disturbance) in V. Solid line: Simultaneous step increase in L ("perfect operator"). Dashed line: Feedback where L is used to control  $x_D$ (PI-settings:  $k = 60, \tau_I = 3.6 \text{ min}$ )



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### Myth about slow control Two-point control



Figure 13: Two-point control: Setpoint change in  $x_D$  from 0.99 to 0.995 with  $x_B$  constant. Solid line: Simultaneous step increase in L and V to their new steady-state values ("perfect operator"). Dashed line: Feedback control using the LV-configuration with PI-settings in (82).



### Myth about slow control Two-point control



Figure 13: Two-point control: Setpoint change in  $x_D$  from 0.99 to 0.995 with  $x_B$  constant. Solid line: Simultaneous step increase in L and V to their new steady-state values ("perfect operator"). Dashed line: Feedback control using the LV-configuration with PI-settings in (82).



# Myth about slow control

Conclusion:

- Experience operator: Fast control impossible
  - "takes hours or days before the columns settles"
- BUT, with feedback control the response can be fast!
  - Feedback changes the dynamics (eigenvalues)
  - Requires continuous "active" control
- Most columns have a single slow mode (without control)
  - Sufficient to close a single loop (typical on temperature) to change the dynamics for the entire column



## **Advanced control**



May also add feedforward control from F (ratio V/F in this case)



#### Advanced control....

Case with valuable bottom product, x<sub>B.</sub> Split range control (SRC):

- 1. Normally control  $x_B$  with boilup V
  - .... But boilup V may reach constraint
- 2. First let reflux L take over
- 3. Then cut back on feedrate F if top reaches constraint



"Systematic Design of Active Constraint Switching Using Classical Advanced Control Structures". A. Reyes-Lua and S. Skogestad, Ind. Eng. Chem. Res. 2020, 59 (6), 2229-2241.



### **Conclusion distillation control**

- Not as difficult as often claimed
- LV-configurations recommended for most columns
- Use log transformations to reduce nonlinearity
- Use composition estimators based on temperature
- Usually: Close temperature loop (P-control OK)
- May use MPC if strong interactions between loops



Chemical Engineering Research and Design (Trans IChemE, Part A), 85 (A1), 13-23 (2007).

S. Skogestad, "The dos and don'ts of distillation columns control",

# Conclusion

- 1. Distillation as a separation process
  - In spite of claims to the contrary it's an efficient process
     it's the heat integration that may be inefficient
  - Unbeatable for high-purity separations
- 2. Modelling
  - In principle it's simple
  - Normally use equilibrium stage model
  - The thermodynamics (VLE) are the most important
- 3. Dynamics
  - There is one dominant slow (drifting) mode
  - related to the holdup of light and heavy key components inside the column
- 4. Optimal operation
  - Minimize energy usage (V) and maximize recovery of valuable component (J = pF F pD D pB B + pV V)
  - subject to satisfying purity specifications
  - Always active constraint: Purity of valueable product («avoid give-away»)
- 5. Control
  - Fist: stabilize the column (levels, pressure and one temperature)
    - The temperature loop will speed up the slow mode, break interactions and provide indirect level control
  - Next: Control active constraints and keep operation close to optimal
  - May use MPC, but can usually do OK with advanced single-loop control

#### https://folk.ntnu.no/skoge/distillation/

