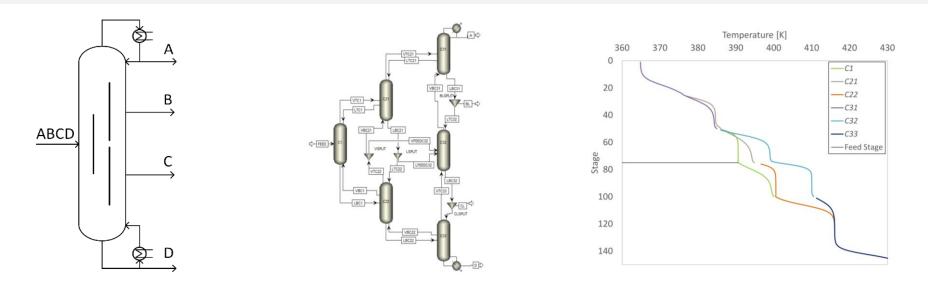






# How to Start up Multiple Dividing Wall Columns – A Theoretical and Experimental Study



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### Introduction

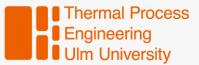


### Distillation

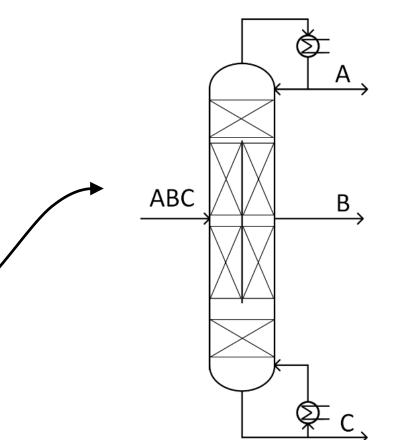
- Biggest share in the energy consumption of the chemical industry
- Low energy efficiency
- High operating costs
  - Energy savings necessary,
    especially in the face of a greener
    economy and rising energy prices!

Multi-component mixtures: separation traditionally in column sequences **Direct split** Indirect split Ð AB Β. ABC ABC C1 C2 C2 C1 ⊊ <sub>BC</sub>∣ ¢ε ¢ε چ B

### Introduction



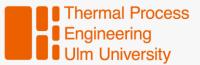
### Dividing Wall Column (DWC)



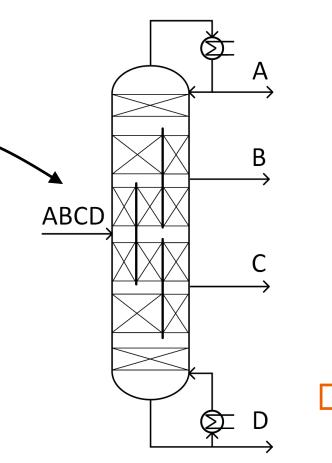
- Separation of ternary mixtures
- Savings of up to 30 % in OPEX and CAPEX<sup>1</sup>
- High complexity of design, operation and control
- First industrial implementation in 1985 by BASF
- Today established technology
- Hundreds of columns already in operation<sup>2</sup>

<sup>1</sup> N. Asprion, G. Kaibel, Dividing wall columns: Fundamentals and recent advances, Chemical Engineering and Processing: Process Intensification 49 (2010) 139–146. <sup>2</sup> H.-M. Lorenz, D. Staak, T. Grützner, and J.-U. Repke, Divided Wall Columns: Usefulness and Challenges, Chemical Engineering Transactions, vol. 69, pp. 229-234, Oct. 2018.

### Introduction



### Multiple Dividing Wall Column (mDWC)



- Separation of mixtures with four or more components
- Savings of up to 55 % in OPEX<sup>3</sup> and lower CAPEX
- Very high complexity of design, operation and control
- No previous practical implementations of mDWCs
  - World's first mDWC comissioned in fall 2021 at Ulm University
  - Research on the whole development cycle of mDWCs
- Meanwhile: world's first commercial application of a mDWC in 2023 at BPCL in Mumbai, India<sup>4</sup>
  - Increasing relevance of mDWCs
  - Need for better understanding on operation including the start-up process

<sup>3</sup> I. Dejanović et al., Designing four-product dividing wall columns for separation of a multicomponent aromatics mixture, Chemical Engineering Research and Design 89 (2011) 1155–1167.

<sup>4</sup> DWC Innovations, Successful Commissioning of World's First Commercial Application of Dual Dividing Wall Column at BPCL, Mumbai, Press Release (2023), https://www.dwcinnovations.com/press-release/successful-commissioning-of-dual-dividing-wall-column-at-bpcl-mumbai/

## Start-up of distillation columns

#### Why is the start-up of distillation columns relevant?

- Start-up generally a non-productive process
- Growing importance of minimizing production costs and generated waste

Achieved by shortening the start-up time

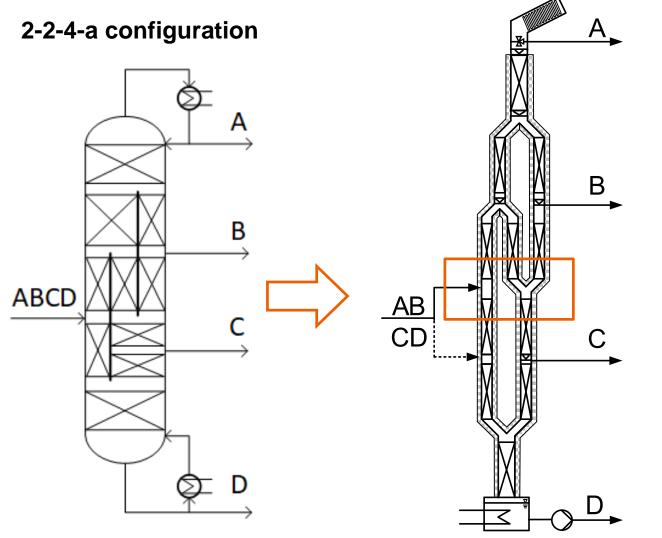
- Intensively discussed topic in research:
  - Several studies on the time-optimal start-up of distillation columns
  - Start-up of dividing wall columns discussed in some works
    - Start-up time strongly dependent on selected start-up strategy
  - Start-up of mDWCs:

> Neither been studied in theory, nor in practice yet, to the best of the author's knowledge

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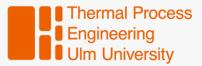
# Pilot column





- Thermally coupled packed column
- Every separation section as separate column
  - Easier construction
  - > No heat transfer across dividing walls
- Total height of 9.6 m
- Column diameter DN80 and DN50 for parallel sections
- Controllable liquid splits
- More than 80 sensors for temperature, pressure (difference) and flow rate

# Pilot column



Thermally coupled packed column

 $\succ$  Easier construction

Controllable liquid splits

(difference) and flow rate

• Total height of 9.6 m

sections

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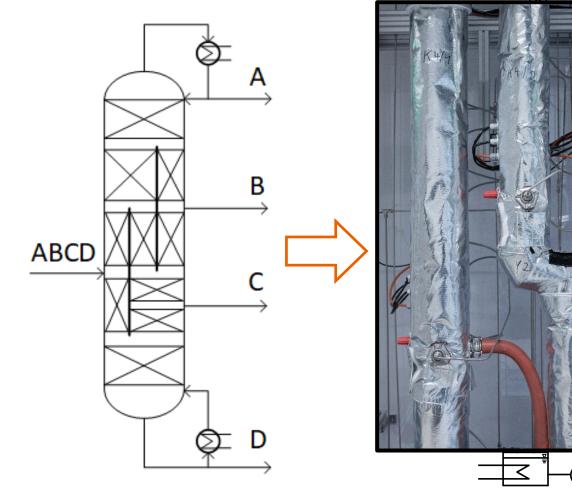
Every separation section as separate column

Column diameter DN80 and DN50 for parallel

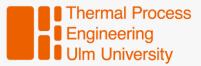
• More than 80 sensors for temperature, pressure

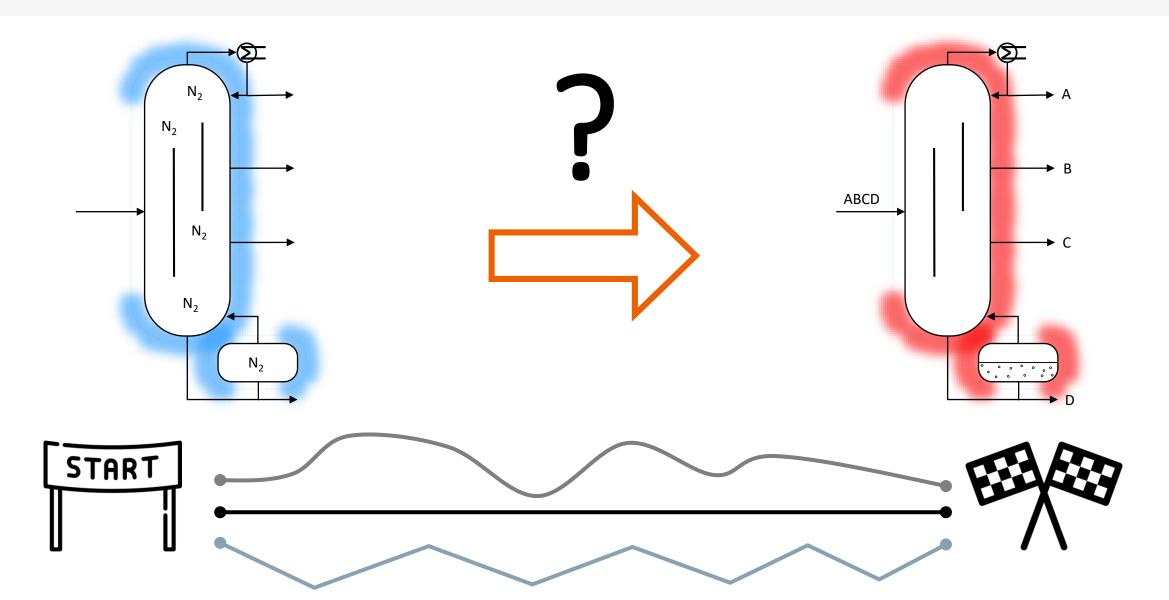
> No heat transfer across dividing walls

#### 2-2-4-a configuration



### Column Start-up







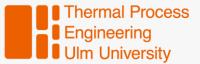
### Development of a start-up strategy using a dynamic simulation in Aspen HYSYS V11

- Equilibrium stage model
- Pressure and gravity driven
- Start-up from dry and cold state

- Test system: Ethanol / n-propanol / i-butanol / n-butanol
- NRTL as thermodynamic model
- Heat capacity of the column and the packing can not be

considered

### New start-up strategy



- No strategies for the start-up of mDWCs available
  - Adaptation of existing start-up strategies for DWCs<sup>5,6</sup>

**Continuous start-up strategy** 

- Developed by Niggemann, Hiller and Fieg<sup>5</sup>
- Continuous feed stream
- Start of heating as reboiler filled to desired level
- All process parameters set to their steady state values when reflux stream sufficient

#### **Discontinuous start-up strategy**

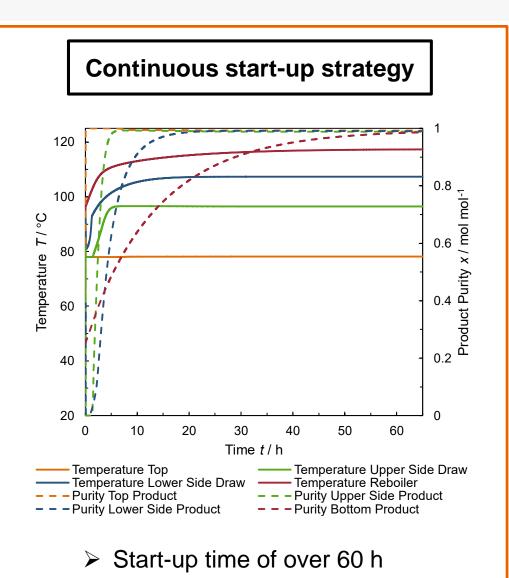
- Developed by Wu, Shen and Ling<sup>6</sup>
- Discontinuous feed stream
- Product withdrawal in intervals
- Constant introduction of feed as steady state temperature profile obtained
- All process parameters set to their steady state

#### values

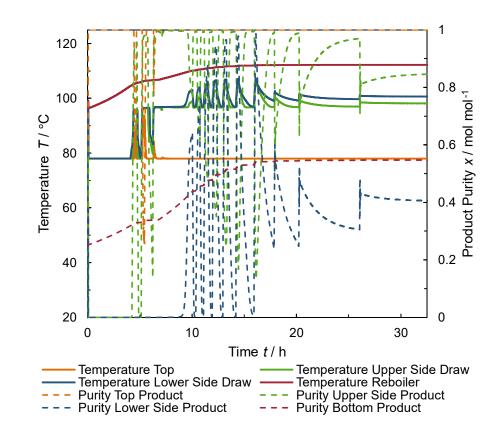
<sup>&</sup>lt;sup>5</sup>G. Niggemann, C. Hiller, G. Fieg, Modeling and in-depth analysis of the start-up of dividing-wall columns, Chemical Engineering Science 66 (2011) 5268–5283. <sup>6</sup>H. Wu, B. Shen, H. Ling, Startup of divided wall column for high purity separation of aromatic reformate, Can. J. Chem. Eng. 96 (2018) 2627–2637.

### Simulation of the start-up process



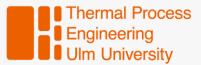


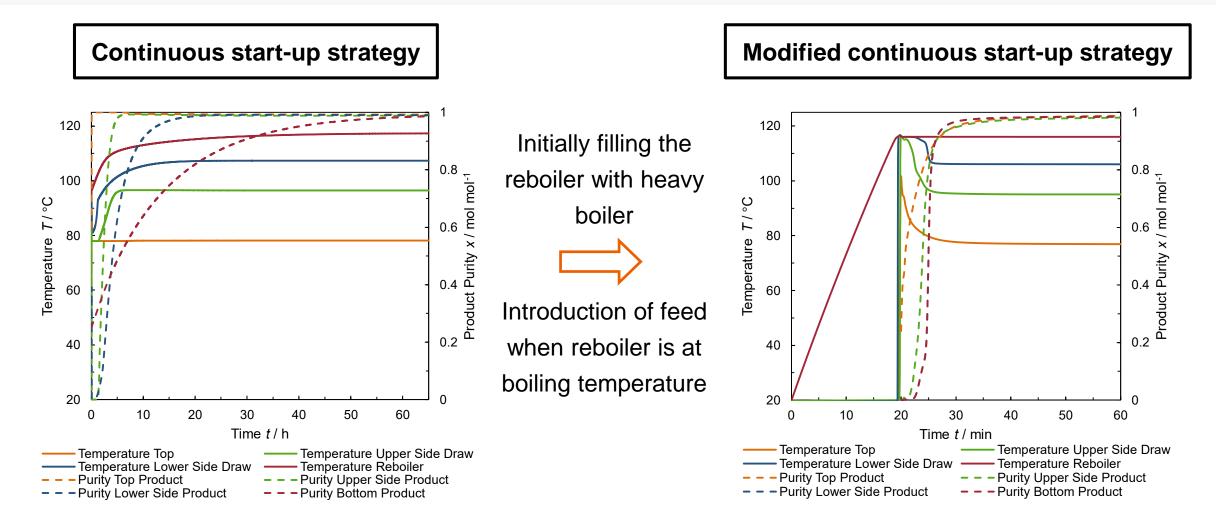
#### Discontinuous start-up strategy



#### Not feasible for the given operating conditions

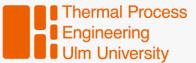
### Simulation - Adjustment of the start-up process



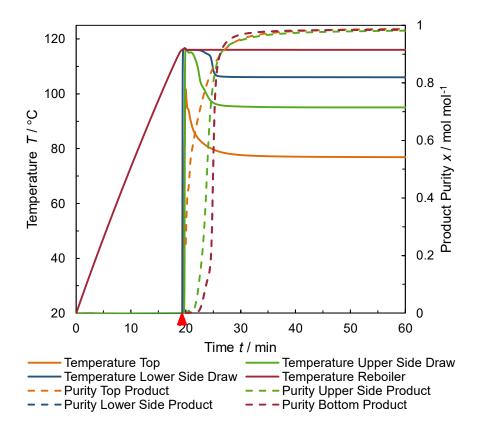


Reduction of the start-up time from over 60 h to considerably shorter time

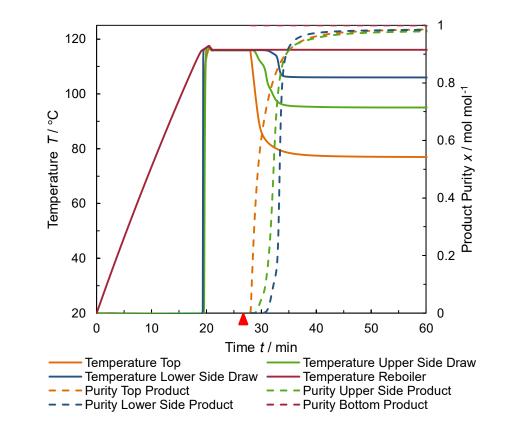
## Simulation - Influence of the time of feed introduction



### When reboiler at boiling temperature

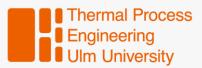


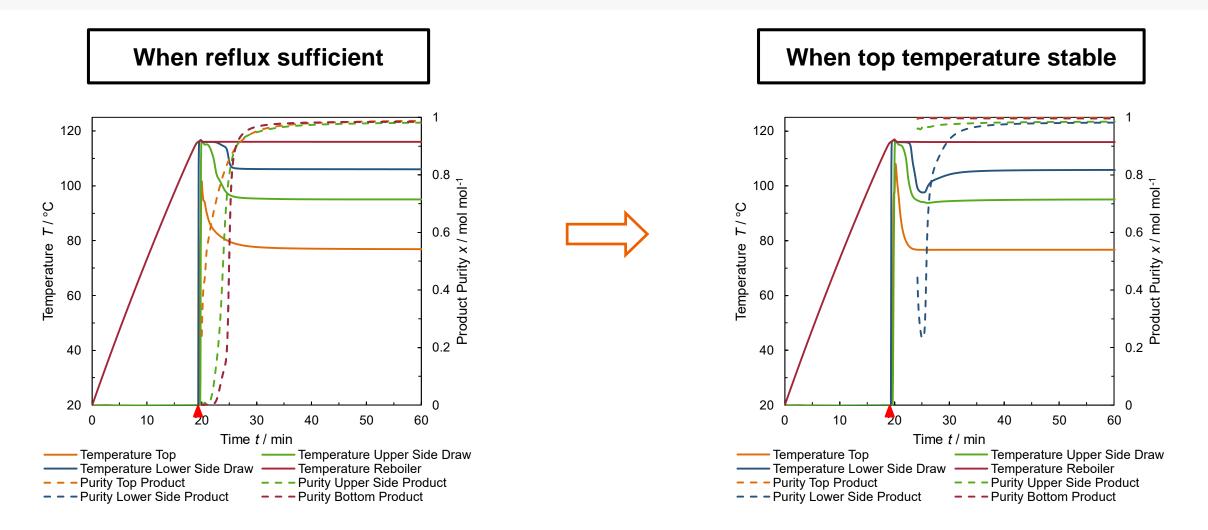
#### When top temperature stable



Delay of the start-up process for later feed introduction

# Simulation - Influence of the time of product valve opening

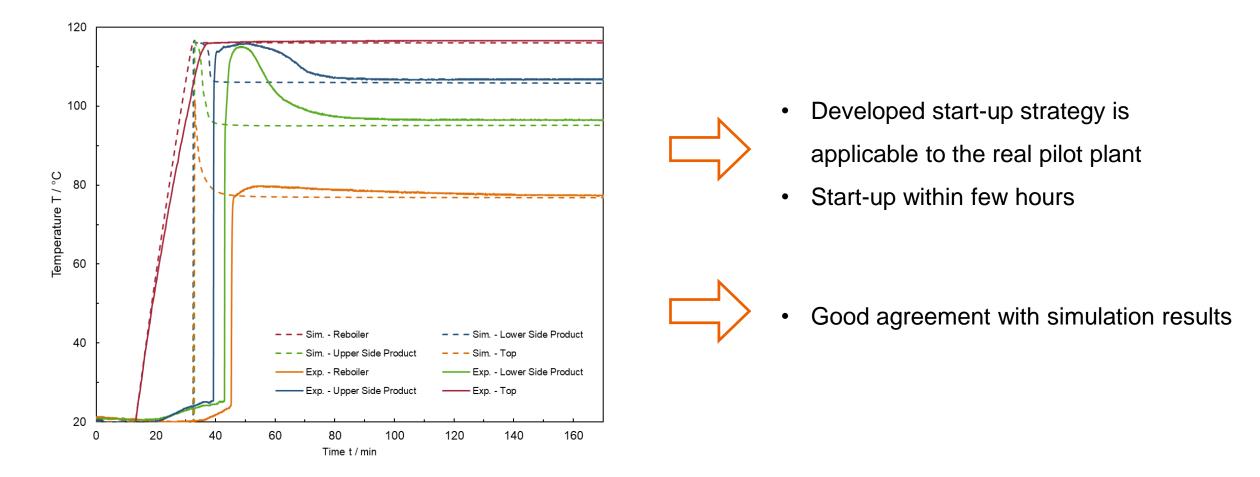




Significant disturbances in the temperature profile for later opening of the product valves

## Experimental validation of the simulation results

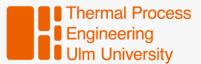
Start-up process of the column with the modified continuous start-up strategy:



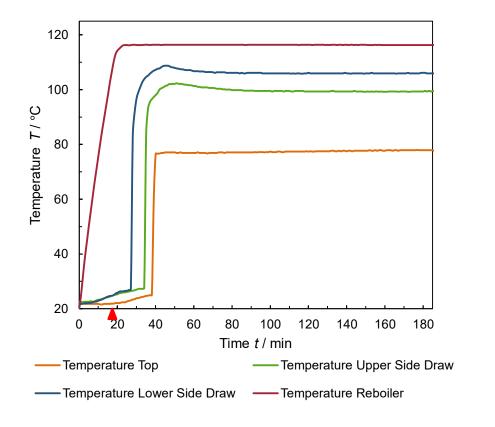
**Thermal Process** 

Engineering

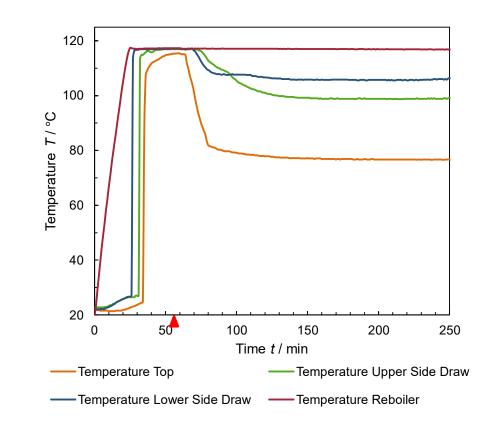
### Experiment - Influence of the time of feed introduction



### When reboiler close to boiling temperature

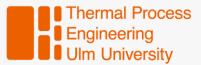


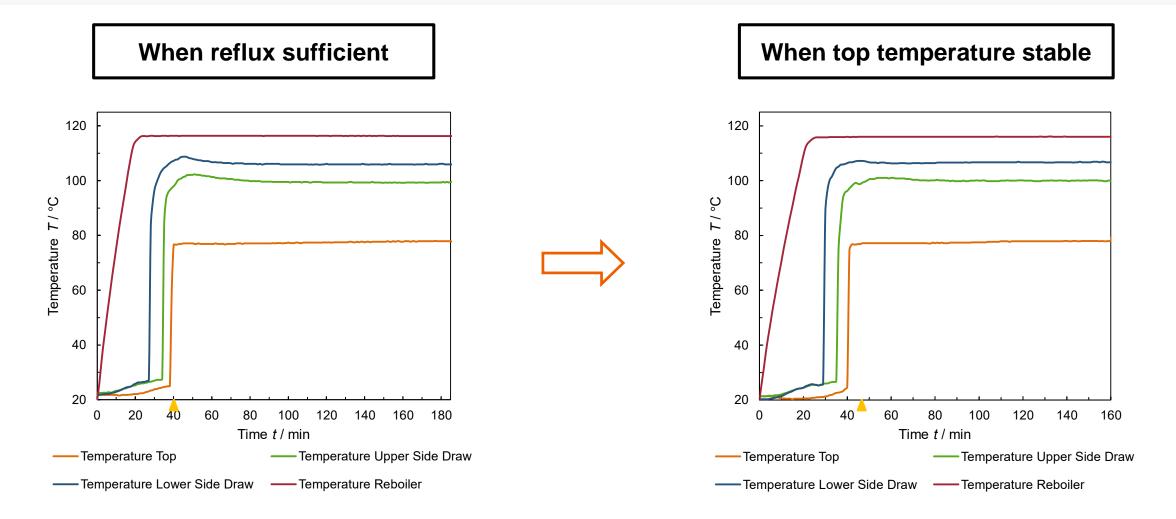
#### When top temperature stable



Delay of the start-up process for later feed introduction

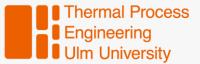
# Experiment - Influence of the time of product valve opening



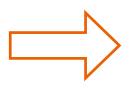


 $\succ$  No large influence of the time of product valve opening, in contrast to the simulation X

### Summary



- World's first multiple dividing wall column comissioned in fall 2021 at Ulm University
- Implementation as thermally coupled 2-2-4-a configuration
- Testing of new start-up strategies for multiple dividing wall columns in a dynamic simulation model
- Development of start-up strategies is heavily based on empirics
- Experimental validation of the simulation model
  - Start-up process in the range of few hours
  - Good agreement with simulation results



Developed start-up strategy allows for a quick and

reliable start-up







# Thank you for your attention!

### Funded by

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**Distillation tailor-made** 

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