Title: Process Intensification In Real Life

Jeffrey FELIX,  | Sulzer Chemtech Ltd. | Process Technology
Presentation overview

- Introduction of Sulzer Chemtech Process Technology
- Process Intensification by combining unit operations
- Concrete PI examples with pervaporation, distillation & crystallization technology
Sulzer Chemtech Ltd.
Leadership in Products & Applications

**Structured packing**
- Tray Technology

**Separators**
- Demisters

**Shell Alliance**

**Random packing**

**Mass Transfer Technology**

**Maintenance, revamps & installation**

**Tower Field Services**

**Process Technology**

**Crystallization Technology**

**Membrane Technology**

**Hybrid Process Plants**

**Film Evaporation**
- L/L-Extraction
- Reactive Distillation

**Multi-component dosing, mixing and application systems**

**Mixpac Systems**
Process Technology
Global organization

PT locations
PT potential expansions
PT presence

USA
Regional headquarters NSA

Canada

Switzerland
Global a. regional headquarters EMA/NSA

Russia

China

Singapore
Regional headquarters APA

Brazil

Netherlands

India

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## Process Technology

### Core activities and markets

<table>
<thead>
<tr>
<th>Technologies</th>
<th>Extraction, distillation, crystallization, membrane, evaporation, absorption, polymer &amp; reaction technology</th>
</tr>
</thead>
<tbody>
<tr>
<td>Segments</td>
<td>Fine &amp; specialty chemical, pharmaceutical process industry, food industry, polymer production</td>
</tr>
</tbody>
</table>
| Products and services | - Engineering services (conceptual, basic & detail)  
                        - Customer testing, process validation, production of samples, toll production  
                        - Process equipment, modular plants, skids  
                        - Solvent recovery  
                        - Product purification  
                        - Hybrid processes and solutions  
                        - Temperature sensitive separations  
                        - Polystyrene, PLA, EPS  
| Process applications | - Solving complex and difficult separation problems  
                        - Supplying advanced, highly efficient process equipment and complete plants  
                        - Extensive experience in combining unit operations |
| Key success factors | - Solving complex and difficult separation problems  
                        - Supplying advanced, highly efficient process equipment and complete plants  
                        - Extensive experience in combining unit operations |
Unique experience in unit operations

- Evaporation
  - Falling film (FFE)
  - Wiped thin film (TFE)
  - Short path (SPE)

- Distillation / rectification
  - Structured packing
  - Random packing
  - Trays

- Liquid / liquid extraction
  - Static columns
  - Stirred columns

- Fractional crystallization
  - Falling film
  - Static system
  - Suspension crystallization

- Membrane systems
  - Pervaporation / vapor permeation
  - Pressure driven membrane solutions
Process Technology
Development competence

- **Process Engineering Studies**
  - Conceptual design
  - Process optimizations / debottlenecking
  - Process synthesis, development and simulation

- **Test Centre**
  - Standard laboratory and pilot equipment for development, pilot and toll processing
  - Testing and validation of distillation, absorption, crystallization, liquid-liquid extraction, film evaporation and membrane processes as well as reaction, devolatilization and mixing tests for polymeric systems

- **Process- & Basic Engineering**
  - Process design, simulation, PFD
  - P&ID, Basic Engineering
Process Technology
Hybrid Approach

- Screening of possible unit operations
  - Experience / reference units / plants
  - In house know how
  - Literature / databases
  - Process simulation
  - Bench scale feasibility tests for promising unit operation

- Combination of unit operations to most cost effective hybrid process

- Validation
  - Process simulation
  - Pilot scale tests / sample generation

- Basic Engineering
- Detail Engineering
- Supply of (key)equipment or skid mounted unit
- Commissioning of turn key hybrid system
Hybrid Processes
Features of specific unit operations

Distillation
- Robust process with high mass transfer rates in liquid and vapor phase
- Phase separation is rapid and complete

Crystallisation
- Separation at low (melting) temperatures
- Separation of mixtures with close boiling components
- Very high specific purification is possible

Liquid-Liquid-Extraction
- Low temperature selective separation @ at mostly ambient pressure
- High throughput @ low energy consumption

Vapour permeation / Pervaporation
- Independent of vapor–liquid equilibrium
- Direct solvent dehydration of azeotropics and close-boilers without entrainer

Pressure driven membrane (UF/NF/RO)
- No phase transition leads to low energy demand
- Separation based on molecule size
Hybrid Processes and application examples

Reactive Distillation

Combine Reaction and Separation in **ONE** Column

**Reactive Distillation Processes**

- **Acetate Technology**
  - Synthesis of Methyl, Ethyl & Butyl Acetate
  - Hydrolysis of Methyl Acetate

- **Acetalisation**
  - Synthesis of Methylal
  - Removal methanol from Formaldehyde

- **Fatty Acid Esters**
Hybrid Processes
Crystallization and Distillation I

Separation of a binary mixture with eutectic and azeotrope
Separation of a binary mixture with an eutectic
Hybrid Application Examples
Crystallization and Distillation

KEY APPLICATIONS
- (ultra) high purities
- color / odor removal
- isomer separation

APPLICATION EXAMPLES
- Dichlorobenzene Isomer Separation
- Nitrochlorobenzene Isomer Separation
- Nitrotoluene Isomer Separation
- Chlorotoluene Isomer Separation
- Meta-Xylene Purification with Ethylbenzene as byproduct
- MDI Isomer Separation and Purification
- Bisphenol A Purification
- Caprolactam Purification
- DMT Purification
- Trioxane
- Monochloroacetic Acid
- High Purity Benzoic Acid
- Glacial Acrylic Acid
- Glacial Lactide
- Naphthalene Purification
- Para-tert. Butylphenol Purification
- Anthracene/Carbazole Separation and Purification
Hybrid processes Crystallization and Distillation
Application example

Lay-out before extension
Hybrid processes Crystallization and Distillation
Application example

Expansion scenario’s
Hybrid processes Crystallization and Distillation  
Pro’s & con’s: why melt-crystallization?

**DISTILLATION**
- general applicable (if volatile)
- proven technology
- engineering correlations available
- known technology at customer
- lab & pilot units available

However
- high operating temperature (boiling point >> degradation)
- energy intensive
- limited purity (VLE depending)
- isomer separation difficult
- close-boilers difficult to separate

**CRYSTALLIZATION**
- applicable for product
- proven, but ‘innovative’ technology
- confirmation pilot tests needed
- new technology for customer
- lab & pilot units available

- lower operating temperature (melting-point >> no side reactions)
- lower energy consumption
- high purity (limited by eutectic point)
- no problem with isomer, close boilers or azeotropic separation
Summary:

- Yield for crystallization of impure mixtures is poor due to phase diagram limitation of the product system

- Crystallization from impure mixtures requires multistage operation that leads to larger equipment and higher energy demand

- Distillation can, at relatively low energy demand, separate the bulk of impurities easily

- Combining the strong points leads to an ideal basis for a hybrid solution and a lower investment @ lower energy consumption

RESULT: **HYBRID OPTION CHosen AND INSTALLED**
Debottlenecking (Base Case*)
Isopropanol Entrainer (azeotropic) Distillation

*base model system = process as modeled by Sulzer Chemtech with data from customer © Sulzer Chemtech Ltd. 2010, all rights reserved – protection notice ISO 16016 – JeF
Energy Efficient Hybrid System
(existing setup with low investment integrated PERVAP™)

Sulzer Chemtech

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** compared to process as modeled by Sulzer Chemtech with data from customer

**觉悟** compared to process as modeled by Sulzer Chemtech with data from customer
Isopropanol recovery
Utility overview

<table>
<thead>
<tr>
<th>Utility costs (per ton product)</th>
<th>Cost Base</th>
<th>original</th>
<th>with integrated PV</th>
</tr>
</thead>
<tbody>
<tr>
<td>Steam (average)</td>
<td>25 €/ton</td>
<td>100 %</td>
<td>55 %</td>
</tr>
<tr>
<td>Cooling Water</td>
<td>0,8 €/m³</td>
<td>100 %</td>
<td>49 %</td>
</tr>
<tr>
<td>Total</td>
<td></td>
<td>100 %</td>
<td>52 %</td>
</tr>
</tbody>
</table>

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Isopropanol recovery

Summary

- Entrainer volume can be reduced by more than 60%
- Capacity increase up to 35% possible
- Energy savings of 120 € per ton/hr of product:
- Annual operation costs PV: ~200’000 Euro/yr
- Simple payback for investment of complete pervaporation system: 2 – 3 years including site engineering, civil engineering & works and pervaporation skid
Tetrahydrofuran (THF)
Base Case: Pressure Swing Distillation

Recycle 3800 kg/h 11.5% H2O

Azeotrope 7'300 kg/h, 6% H2O

Feed
10'000 kg/h
35 % THF
65 % H2O

Atmospheric Column T-1

Water 6'500 kg/h

Pressure column T-2
(8 bar)

Product 3'500 kg/h
> 99.97 % THF

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Tetrahydrofuran (THF)
Pressure Swing Distillation with Pervaporation Unit

**Feed**
10'000 kg/h
35 % THF
65 % H2O

**Atmospheric Column T-1**
3'785 kg/hr
0.9 % H2O

**Recycle**
285 kg/h
11.5 % H2O

**Azeotrope**
3'985 kg/h, 5.8 % H2O

**Pervaporation Unit**
3'785 kg/hr
> 99.97 % THF

**Permeate**
200 kg/h, >90% H2O

**Water**
6'500 kg/h

**Pressure column T-2**
(8 bar)

**Product**
3'500 kg/h
> 99.97 % THF
Tetrahydrofuran (THF)
Comparison Heat Balances

<table>
<thead>
<tr>
<th>Without PV</th>
<th>With PV</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Steam 4 bara</strong></td>
<td><strong>Steam 4 bara</strong></td>
</tr>
<tr>
<td>Reboiler T-1</td>
<td>1.78</td>
</tr>
<tr>
<td>Reboiler T-2</td>
<td>1.22</td>
</tr>
<tr>
<td><strong>Reboiler T-1</strong></td>
<td>1.78</td>
</tr>
<tr>
<td><strong>Reboiler T-2</strong></td>
<td>1.22</td>
</tr>
<tr>
<td>PV Stage</td>
<td>0.20</td>
</tr>
<tr>
<td><strong>Total steam</strong></td>
<td>3.00</td>
</tr>
<tr>
<td><strong>Cooling water</strong></td>
<td><strong>Cooling water</strong></td>
</tr>
<tr>
<td>Cond. T-1</td>
<td>1.33</td>
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<tr>
<td>Cond. T-2</td>
<td>0.91</td>
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<tr>
<td>Perm. Cond.</td>
<td>0.14</td>
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<tr>
<td><strong>Total CW</strong></td>
<td>2.24</td>
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</table>

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<th>With PV</th>
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<tbody>
<tr>
<td><strong>Steam 4 bara</strong></td>
<td><strong>Steam 4 bara</strong></td>
</tr>
<tr>
<td>Reboiler T-1</td>
<td>3.01</td>
</tr>
<tr>
<td>Reboiler T-2</td>
<td>2.05</td>
</tr>
<tr>
<td><strong>Reboiler T-1</strong></td>
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<tr>
<td><strong>Reboiler T-2</strong></td>
<td>2.05</td>
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<tr>
<td>PV Stage</td>
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<tr>
<td><strong>Total steam</strong></td>
<td>5.06</td>
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<tr>
<td><strong>Cooling water</strong></td>
<td><strong>Cooling water</strong></td>
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<tr>
<td>Cond. T-1</td>
<td>114</td>
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<tr>
<td>Cond. T-2</td>
<td>78</td>
</tr>
<tr>
<td>Perm. Cond.</td>
<td>12</td>
</tr>
<tr>
<td><strong>Total CW</strong></td>
<td>192</td>
</tr>
</tbody>
</table>

- Decrease energy consumption or increase capacity with pervaporation
- Debottleneck and save energy within the same step

Production 28.000 t/year > 99.97 wt% THF
Thank you for your attention!
Questions?

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Process Intensification by providing superior and competitive hybrid separation technology solutions with guaranteed performance