Verfahrenstechnische Anlagen GmbH
Company introduction

Thermal separation with thin film and short path distillation

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Company introduction

MAX STREICHER GmbH & Co. KG aA
- Road Construction and Civil Engineering
- Bridge Building and Structural Engineering
- Building and Industrial Construction
- Pipeline and Plant Construction
- Communications Networks

STREICHER Group
Employees: 2,300
Turnover: ~250 Million €
VTA Products and services:

- Wiped film evaporators and plants
- Short path evaporators and plants
- Sludge drying plants
- Process development, laboratory and pilot trials
- Toll distillation with wiped film and short path evaporators
Company introduction

VTA GmbH with manufacturing facilities of STREICHER Maschinenbau GmbH
Basic background for Wiped Film and Short Path Evaporation

Batch distillation

- Discontinuous process
- Very long residence time
- Higher temperature necessary for evaporation due to liquid level.

Example:

\[ h = 2 \text{ m} \]
\[ \rho = 800 \text{ kg/m}^3 \]
\[ P_{\text{pot}} = 200 \text{ Pa} = 2 \text{ mbar} \]
\[ P_{\text{heating}} = 15.896 \text{ Pa} = 160 \text{ mbar}. \]

\[ P_{\text{heating}} = P_{\text{pot}} + \rho \times g \times h \]
Low operating pressures reduce the thermal decomposition of the product during distillation.
Basic background for Wiped Film and Short Path Evaporation

Thermal decomposition of product can lead to:

- polymerization of valuable component
- change of viscosity of final product
- solid forming during the evaporation process
- product loss due to lower product yields
- decrease of odor and taste quality (food application)
- Decolorization of the product

This is why it is important to reduce the operating temperature and the hold up time during evaporation.
Basic background for Wiped Film and Short Path Evaporation

Natural or forced circulation evaporator

- long residence time
- poor vacuum
- circulated liquid is at the final concentration
- low concentration gradient

1 feed
2 distillate
3 residue
4 heating
5 cooling
6 vacuum
Basic background for Wiped Film and Short Path Evaporation

**Task for heat sensitive products:**
Reduce product liquid level to a thin film

**FILM EVAPORATION**

Heating media

Product film

$h \sim 1 \text{ mm}$
Basic background for Wiped Film and Short Path Evaporation

Falling Film Evaporator
- no liquid column
- longer residence time due to circulation of product
- pressure drop
- limited flexibility
- very sensitive to fouling
- circulated product is high concentrated
- Best for removal large quantity of lights (water or solvent)
Basic background for Wiped Film and Short Path Evaporation

Target for heat sensitive products in addition to film evaporation:
Reduction of hold up time during evaporation

Requirement:
Rapid evaporation

Therefore necessary:
High turbulence in the film- possible by wiping the film
Basic background for Wiped Film and Short Path Evaporation

Non wiped film

Heating media

Product film

$C_1, T_1$

$C_2, T_2$
Basic background for Wiped Film and Short Path Evaporation

Wiped film

- Turbulent film
- Wiper element
- Bow wave
- Heating media
- Product film
Basic background for Wiped Film and Short Path Evaporation

wiped film

Heating media

Product film

\[ C_1, T_1 \quad C_2, T_2 \]
Basic background for Wiped Film and Short Path Evaporation

Wiped film evaporator
Increase of turbulence
• increased heat transfer by turbulent film
• short residence time
• wiping of viscous and solidified films possible
• achievable pressure 1 mbar
Basic background for Wiped Film and Short Path Evaporation

- Wiper drive
- Vapor nozzle
- Heating jacket
- Residue outlet
Basic background for Wiped Film and Short Path Evaporation

Reduction of pressure drop:
Example: Volume expansion of water at vacuum.

1 l water evaporated:
volume = 1 m³ - pressure = 1000 mbar
volume = 1.000 m³ - pressure = 1 mbar
volume = 1.000.000 m³ - pressure = 0,001 mbar
Evaporator heating jacket surface area, example 3 m²

In case of a wiped film evaporator the vacuum nozzle has a diameter of 300 mm.

PRESSURE DROP CAUSED AT LOW OPERATING PRESSURES
Wiped film evaporator limited to operating pressures of 1 mbar
Basic background for Wiped Film and Short Path Evaporation

Heating media

Cooling media

Product film
distillate
Basic background for Wiped Film and Short Path Evaporation

Short Path Evaporator

- short residence time due to turbulent film
- very small pressure drop for vapors
- achievable pressure \( 10^{-3} \text{ mbar} \)

but:
- limited viscosities, no solidifying products distillable
Basic background for Wiped Film and Short Path Evaporation

condenser

Heating jacket

Vacuum nozzle
Basic background for Wiped Film and Short Path Evaporation

Wiper basket

Heating jacket
Basic background for Wiped Film and Short Path Evaporation

Industrial wiped film evaporation plants consist in

• Feeding system
• Product preheating
• preflashing stage (necessity depending on process cond.)
• Wiped film evaporator
• continuous concentrate discharge
• External condenser
• continuous distillate discharge
• Chilled post condenser (depending on vacuum system)
• Vacuum system
Basic background for Wiped Film and Short Path Evaporation

Wiped film evaporation flow sheet
Basic background for Wiped Film and Short Path Evaporation

Industrial short path evaporation plants consist in:
- Feeding system
- Product preheating
- Degassing stage (pressure 1 - 10 mbar)
- Short path evaporator with internal condenser
- Continuous concentrate discharge
- Continuous distillate discharge
- Chilled post condenser (cold trap)
- Vacuum system
Basic background for Wiped Film and Short Path Evaporation

Short path evaporation flow sheet
Evaporator sizes

Wiped film evaporators

<table>
<thead>
<tr>
<th>Thinfilm Evaporators</th>
<th>Surface [m²]</th>
<th>Height [mm]</th>
<th>Diameter [mm]</th>
<th>Height for Disassembly [mm]</th>
</tr>
</thead>
<tbody>
<tr>
<td>VD 70-6</td>
<td>0.06</td>
<td>0.4</td>
<td>70</td>
<td>0.8</td>
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<tr>
<td>VD 125-20</td>
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<td>VD 2600-8000</td>
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</table>
Evaporator sizes

Wiped film evaporator:

Conical wiper
## Short path evaporators

<table>
<thead>
<tr>
<th>Type</th>
<th>Surface [m²]</th>
<th>Height [mm]</th>
<th>Diameter [mm]</th>
<th>Height for Disassembly [mm]</th>
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<tbody>
<tr>
<td>VK 70-6</td>
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<td>VK 125-15</td>
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<td>5.550</td>
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<tr>
<td>VK 2000-3800</td>
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<td>9.610</td>
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<td>2,000</td>
<td>23.260</td>
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Evaporator sizes

Short path evaporator:
Evaporator sizes

Horizontal evaporators

<table>
<thead>
<tr>
<th>Typ Type</th>
<th>A [m²]</th>
<th>L [mm]</th>
<th>d[m] [mm]</th>
<th>L^[*] [mm]</th>
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<tbody>
<tr>
<td>VDC/H 110-10</td>
<td>0.10</td>
<td>1.300</td>
<td>110</td>
<td>500</td>
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<td>VDC/H 260-50</td>
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<td>2.750</td>
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<td>1.250</td>
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<td>VDC/H 370-100</td>
<td>1.00</td>
<td>3.450</td>
<td>370</td>
<td>1.590</td>
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<tr>
<td>VDC/H 530-200</td>
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<td>4.000</td>
<td>530</td>
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<tr>
<td>VDC/H 630-400</td>
<td>4.00</td>
<td>5.100</td>
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<td>2.600</td>
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<td>VDC/H 750-650</td>
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<td>750</td>
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<td>VDC/H 800-1200</td>
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<td>7.200</td>
<td>800</td>
<td>4.800</td>
</tr>
</tbody>
</table>
Evaporator sizes

Horizontal evaporators

Conical evaporator with variable gap between rotor and heating jacket

Horizontal evaporator for drying until solid content
Evaporator sizes

Glass lined evaporators

To be applied in case of high corrosive products
max. temperature: 200 °C
max. Evaporator size: 12 m²
Wiper systems

VTA wiper systems

Selection according to product and process requirements

- viscosity
- solid content
- purity
- specific load
Wiper systems

VTA Wiper systems

- Roller Wiper System WRS
- Scraper Block Wiper SKR
- Swing Blade System SBS
- Pendelum Blade System PBS
- Ridged Rotor System RRS
Wiper systems

- for low viscosity (up to 1000 mPa s)
- Rollers in PTFE or Graphite
- high wear resistance
- not usable for polymerizing or solidifying products
Wiper systems

VTA evaporator supply

- Low and higher viscosity (1 - 20,000 mPa s)
- can handle polymerizing products
- influence to residence time by grooves
- centrifugal force or spring plates
Wiper systems

SKR Rotor example
Wiper systems

Comparison of film distribution WRS - SKR

WRS - Wiper

SKR - Wiper
Wiper systems

Movie taken inside a VK 2000-5000 with 50 m² surface and WRS-Wiper
Wiper systems

Movie taken inside a VD 2000-3800 with 38 m² surface and SKR-Wiper
Wiper systems

Rigid rotor or fixed clearance rotor

- fix gap between rotor and evaporator wall
- high viscosity
- bottom bearing required
- not for Short Path design
- Lower distillation rates possible
- special design: cone shape evaporator for variable hold up time
Industrial supply examples

Components and plants for the
- Chemical & polymer industry
- Pharmaceutical industry
- Oleochemical industry
- food ingredient
- cosmetic
- flavor and fragrance production
Industrial supply examples

Chemical & polymer industry: MDI removal from prepolymer
Industrial supply examples

Pharmaceutical industry, production of an API for an US pharmaceutical producer
Industrial supply examples

Components and plants for Oleochemical industry
Industrial supply examples

Evaporation plant applied for the food ingredient industry
Industrial supply examples

Plant applied for the flavor and fragrance production
Process design

Task clarification:
• Information about the feed composition
• Information about the final product quality
• Information about physical properties
  - Safety information (MSDS)
  - Melting point of product fractions
  - Evaporation temperature at relevant pressure
Information about the feed composition:

What is the light volatile content?

Information about the light volatile content can be analyzed by GC or Carl Fisher Titration (determination of water).
<table>
<thead>
<tr>
<th>Process design</th>
</tr>
</thead>
<tbody>
<tr>
<td>Problem in case the light volatile content is too high:</td>
</tr>
<tr>
<td>Flashing of product entering the evaporator at vacuum.</td>
</tr>
<tr>
<td>Example: Evaporation performed in a short path evaporator at 0.1 mbar, 2% of light solvent is present in the feed mixture</td>
</tr>
<tr>
<td>Problem: Residue droplets are dragged to the internal condenser due to flash evaporation</td>
</tr>
<tr>
<td>Increasing of pressure in the evaporation area</td>
</tr>
<tr>
<td>Solution: Flash evaporation prior entering the short path evaporator at moderate vacuum</td>
</tr>
</tbody>
</table>
Process design

What is the physical condition of the
• crude
• distillate
• concentrate
• side fractions
at ambient and operation temperature

Information necessary:
• viscosity
• melting point

Possible consequence:
• necessary trace heating or jacketing of product wetted surfaces
• selection of wiper system
Information about the necessary stages for evaporation:

Depending on the feed composition
• Light volatile fraction
• middle fraction
• heavy fraction
• other fraction

min. necessary temperature difference between each fraction necessary in general:
30 - 40 K,
in case the temperature difference is less,
Fractional distillation has to be selected,
Problem: higher operation pressure necessary- min. 2 - 3 mbar

Example: Fishoil concentrate distillation
Process design

Selection of general process conditions depending on:

- physical properties of the product (boiling point (f) pressure)
- available utilities
- max. allowable temperature of the product

to be considered:
experience often related to long residence times, allowable temperatures in wiped film or short path evaporators are higher
## Process design

**Important considerations:**

- which thermal separation process should be selected
- At what cooling media temperature can the product condense
- What is the most economical cooling media (most preferrable aim: condensation on cooling water based media)
- What heating media can be applied
- Cold trap condensation
Laboratory plants

How to process products on the laboratory plant:

Feed composition clarification
Feed product conditioning
Database research for physical properties
Vacuum test of the laboratory plant
Heating temperature set point adjustment
Vacuum adjustment
cold trap chilling
Degassing step
Evaporation step
Cleaning of the plant
Laboratory plants

VKL 70 Short Path Evaporators:

- VKL 70-4
- VKL 70-4 R
- VKL 70-4 FDR
- VKL 70-4 FDRR
Laboratory plants

VDL 70 Wiped Film Evaporators:

VDL 70-4  VDL 70-4 C  VKL 70-4 R
Laboratory plants

Special sized Glass Evaporators:

VKL 125-15

VKL 38-1

VDL 70-7
Laboratory plants

External condensers:

Standard

Heated condenser jacket
Laboratory plants

Cold traps:

- Dry Ice or liquid \(N_2\)
- Liquid refrigerant
Laboratory plants

Feeding Systems:

- Dosing Vessel
- Membrane Pump
- Gear Pump with feed vessel
Laboratory plants

Feeding Systems, VTA Gear Pump:
Laboratory plants

Discharge Systems:

- Triple Split Distributor
- Intermediate Buffer Vessel
- Discharge Pump
- Carousel
Laboratory plants

Vacuum components:

- Rotary Vane Pumpe
- Combination with oil diffusion Pump
- Pressure Gauge Pressure Adjustment
Laboratory plants

Standard Unit VKL 70-4:
Laboratory plants

Standard Unit VKL 70-4:
Laboratory plants

Standard Unit VKL 38-1:
Laboratory plants

VKL 70-4 with options:

- continuous feeding and discharging pumps
- hot oil unit suitable for temperatures of > 350 °C
- all produce wetted parts in jacketed design
- oil diffusion pump
Laboratory plants

VDL 70 with column:

- increasing No. Of theoretical trays
- Advantage of short hold up time during reboiling
- Able to reboil until viscous and solid residues
Pilot plants

Criteria to select options for the pilot plant (stainless steel)

What are the min. product quantities to be processed during piloting?
- < 20 kg: VD/VK 70 with 0,06 m²
- < 100 kg: VD/VK 125 with 0,15 and 0,2 m²

Will the plant be manually or automatically operated?
- Influence on Instrumentation and controls

What are the melting points of the products?
- Jacketed design of prod. wetted parts to be considered

Is the installation site explosion proved area?
- Clarification necessary: Location of switch and control cabinet
  - Location of heating and chilling units

Further product information:
- Boiling points as (f) of pressure,
- viscosities/solid content of product and side streams,
- number of distillation steps necessary
Pilot plants

Pilot plant VK 70-6 with predegasser
Pilot plants

Pilot plant VD 125-20, VK 125-15 VK 200-40 with control system
Cleaning procedure of pilot plants

- Select cleaning solvents in accordance with the processed product, Polar/unpolar solvents
- Flush the plant with the distillate of the previous processed product
- if possible, operate the trace heating to heat up the cleaning solvent in order to increase the solubility
- In case the processed product formed big solid particles, block out the gear pumps- damage is possible
- from time to time, fill up product piping and equipment with caustic and operate if possible the heating and trace heating
- Rins finally with water and ethanol
- Dry the plant by operating the heating and vacuum
Heating jacket design

Normal double wall jacket
Heating jacket design

Half coil heating jacket
Heating jacket design

Reinforced jacket
Vacuum technology

Pressure range of vacuum pumps

- Liquid ring pump
- Diaphragm pump
- Sliding vane rotary pump
- Roots pump
- Vapor jet pump
- Diffusion pump
- Turbomolecular pump

<table>
<thead>
<tr>
<th>Pump Type</th>
<th>Ultrahighvacuum</th>
<th>High vacuum</th>
<th>Medium vacuum</th>
<th>Rough vacuum</th>
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<tbody>
<tr>
<td>Liquid ring pump</td>
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<tr>
<td>Diaphragm pump</td>
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<tr>
<td>Sliding vane rotary pump</td>
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<tr>
<td>Roots pump</td>
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<td>Vapor jet pump</td>
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<td>Diffusion pump</td>
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<tr>
<td>Turbomolecular pump</td>
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</table>

Pressure range:
- Ultrahighvacuum: $10^{-9}$ mbar
- High vacuum: $10^{-6}$ to $10^{-5}$ mbar
- Medium vacuum: $10^{-3}$ mbar
- Rough vacuum: $10^{0}$ to $10^{-2}$ mbar