Pervaporation and Vapour Permeation Membrane Technology in the Flavour and Fragrances Industry

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- Pervaporation and Vapour Permeation

- Applications in Flavours and Fragrances Industry
  - Winning of Flavours and Fragrances
  - Recovery of solvents
  - Winning of products from fermentation
  - Solvent purification

- Product Portfolio
Pervaporation and Vapour Permeation
Pervaporation

- Pervaporation is the separation of **liquid mixtures** by **partial vaporization** through a **membrane**
- The heat for vaporization is dissipated from the **feed**
Vapour Permeation

- Vapour permeation is the separation of **vapour mixtures** through a **membrane**
- No additional heat is required in the membrane module
### Economics examples PV/VP

<table>
<thead>
<tr>
<th>Application</th>
<th>Conventional process</th>
<th>Energy savings</th>
<th>Costs savings</th>
<th>Pay back period</th>
</tr>
</thead>
<tbody>
<tr>
<td>Bio-ethanol</td>
<td>Distillation + mol sieve</td>
<td>30%</td>
<td>20% (3 €ct/l lower EtOH production cost)</td>
<td>3 years</td>
</tr>
<tr>
<td>Acetone</td>
<td>Distillation</td>
<td>40%</td>
<td>25%</td>
<td>2 years</td>
</tr>
<tr>
<td>Esterification reactions</td>
<td>Distillation (batch process)</td>
<td>50%</td>
<td>30%</td>
<td>± 1 year</td>
</tr>
<tr>
<td>Methanol from toluene</td>
<td>Extraction + distillation</td>
<td>50%</td>
<td>30%</td>
<td>± 1 year</td>
</tr>
</tbody>
</table>

Source ECN 2013
Main Advantages PV/VP

- Flavours & Fragrances recovery with high yield and quality
- Separation of azeotrope mixtures without entrainers
- Low energy consumption
- High purity
- Multi component mixtures with small differences in BP’s can be dehydrated
- The feed mixtures to be treated may be supplied in liquid (PV) or vapour form (VP)
- High degrees of flexibility regarding the feed mixtures (multi-purpose system)
- In situ dehydration of condensation reactions increase the process yield and efficiency
Applications in Flavours and Fragrances Industry
Case:

Flavour and Fragrances winning
Flavour and Fragrances winning

Applications: e.g. fruit aromas, coffee aroma, fragrances

Pervatech units:
  Process: Pilot and full scale manufacturing
  Membranes: PDMS on ceramic tube
  Condensation: multi step fractionated
  Compliance: ATEX / PED 97/23 Pressure Directive
              2006/42 EG Machine Directive
              1935/2004 EG Food Contacting Materials

Advantages: 10x higher concentration than conventional processes
  Highly volatile components will remain
  Flavour composition remains in “natural” balance
  Less sensitive to feed concentration variations than conventional
Case:

Ethanol recovery in Flavour and Fragrances industry
**Ethanol recovery in Flavour and Fragrances Industry**

**Application:** Recovery of solvent used in extractions; removal of undesired organics. Current re-use: 2-3 times.

**Pervatech solution:**

- **Process:** Pervaporation
- **Membranes:** Hybrid silica HybSi®

**Result:**
- Nearly virgin ethanol recovered
- No organoleptic compounds detectable
- Up to 50 times re-use possible

<table>
<thead>
<tr>
<th></th>
<th></th>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>EtOH purchase</td>
<td>900</td>
<td>81.000</td>
<td>4.860</td>
</tr>
<tr>
<td>Transport</td>
<td>150</td>
<td>13.500</td>
<td>810</td>
</tr>
<tr>
<td>Incineration</td>
<td>1.400</td>
<td>126.000</td>
<td>7.560</td>
</tr>
<tr>
<td>Total</td>
<td>2.450</td>
<td>220.500</td>
<td>13.230</td>
</tr>
</tbody>
</table>
Case:

Ethanol recovery from algae
Ethanol extraction – DEMA project (1)

FP7-EU Project DEMA

http://www.dema-ethol.eu/en/

Original Target: 50 g/l EtOH
Revised Target: 10-20 g/l EtOH
Status: 5-6 g/l EtOH
Ethanol extraction – DEMA Project (2)

Pervatech pilot plant:

Membranes: 2 x PDMS
Temperature: max. 80 °C
Circulation flow: 500-3000 L/h
Condensation: 2 step

Feed: Microalgae broth with 1% ethanol
Testing conditions
T: 35-50 °C
Re: 13000
Flux: 3-5 kg/m²h
**Ethanol extraction – DEMA project (4)**

Extraction, concentration and purification to fuel grade, using only membranes

<table>
<thead>
<tr>
<th>Membrane step</th>
<th>x_EtOH_feed [wt. fraction]</th>
<th>x_EtOH_product [wt. fraction]</th>
<th>Membrane type</th>
<th>Separation factor</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>0.01</td>
<td>0.09</td>
<td>PDMS</td>
<td>9</td>
</tr>
<tr>
<td>2</td>
<td>0.09</td>
<td>0.38</td>
<td>PDMS</td>
<td>7</td>
</tr>
<tr>
<td>3</td>
<td>0.38</td>
<td>0.63</td>
<td>PDMS</td>
<td>3</td>
</tr>
<tr>
<td>4</td>
<td>0.63</td>
<td>0.73</td>
<td>PDMS</td>
<td>1.6</td>
</tr>
<tr>
<td>5</td>
<td>0.73</td>
<td>0.995</td>
<td>HybSi® Dense</td>
<td>100-300(^+)</td>
</tr>
</tbody>
</table>

\(^+\)H\textsubscript{2}O/EtOH selectivity, HybSi® is a hydrophilic membrane
Case:

Solvent purification
Azeotropic Distillation IPA
Azeotropic Distillation IPA (1)
Conventional Distillation Process

Eliminate:

i) heating process

ii) use of benzene
Azeotropic Distillation IPA (2)
Hybrid Process

Distillation

Column 1
stages: 10
9th stage
reflux ratio: 1.35

Feed, 1000 kg/h
50 wt.% IPA
50 wt.% water

601 kg/h,
83 wt.% IPA
17 wt.% water

Pervaporation

Pervaporation

Retentate, 496 kg/h
99.59 wt.% IPA
0.41 wt.% water

Permeate, 105 kg/h
95.44 wt.% water
4.56 wt.% IPA

Waste, 399 kg/h
0.37 wt.% IPA
99.63 wt.% water

(a)
Azeotropic Distillation IPA (3)
Cost reduction potential

*Source: Economic comparison between azeotropic distillation and different hybrid systems, Elsevier 2004*

<table>
<thead>
<tr>
<th>Operation Cost</th>
<th>Investment Cost</th>
<th>Maintenance Cost</th>
</tr>
</thead>
<tbody>
<tr>
<td>36.65 €/ton</td>
<td>78.28 €/ton</td>
<td>15.11 €/ton</td>
</tr>
<tr>
<td>17.25 €/ton</td>
<td>42.16 €/ton</td>
<td>12.45 €/ton</td>
</tr>
</tbody>
</table>

Total Cost:
- Distillation: €130/ton
- Hybrid: €72/ton

-45% reduction in cost
Product Portfolio
Product Portfolio (1a) Hydrophilic Membranes

Hybrid Silica HybSi®- AR (acid resistant)

Applications:

- Recovery of solvents; e.g. in Flavour & Fragrances industry
- Breaking of azeotrope
- Removal of water from Organics
  - Alcohols, a-protic solvents, DMAC,
  - DMSO, DMF, Ethyl acetate, NMP, Phenol, THF, ACN …
- In situ dehydration of condensation reactions
  - Esterification, Transesterification
  - Acetalization
  - Ketalization
- Dehydration of essential oils
- Separating lower Mw solvents from higher Mw solvents (MeOH – ACN)
Product Portfolio (2) Organophilic Membranes

PDMS, POMS, PEBA (flat sheet, spiral wound, coated inside ceramic tube)

Applications:

• Winning of aromas, flavours, fragrances
• Removal of “off-flavours”
• Recovery of organics from fermentation broth (e.g. ABE)
• Waste water treatment (at point of release)
• Recovery of aromatic compounds from tank-heads (VOC removal)
4-tube Membrane Assembly

Membrane top layer: Hybrid Silica HybSi®-AR, zirconia, titania, PDMS
Assembly configuration: 4 tubes of 10 x 7 mm (Od x Id)
Assembly dimension: 1200 x 25 mm (0.1 m²) / 600 x 25 mm (0.05 m²) (L x D)
4-tube Membrane Assembly Modules Industrial

Availability: 0.7; 1.9; 3.8; 9.4; 22.5; 48.7 m² modules

(example = 0.7 m²)
Product Portfolio (3)

Products for Research and Application testing

- Test modules
  - 1-Tube
  - 4-Tube
  - Flat Sheet cell

- Lab testers (in EX, non-EX, Food grade)
  - Pervaporation (PV)
  - Vapour permeation (VP)
  - Combi (PV/VP)

- In-house testing
  - Dehydration
  - Concentration
Product Portfolio (4)

Products for industrial installations

- Membrane Module Program Hydrophilic – Organophilic
- Proof of Principle testing
- Pilot Plants (EX, non-EX, Food grade)
- Industrial plants via
  - OEM
  - System integrator
  - End User
Customers / Market Segments

- End-Users
- OEM’s
- Universities / Research Institutes

- Chemical / Pharmaceutical / Food / Flavours & Fragrances

- World wide

- Focus on niche applications
Questions?

Thank you for your attention

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