DEODORIZATION AND PHYSICAL REFINING

Wim De Greyt
De Smet Group   Belgium
Physical versus chemical refining

Direct refining cost

Cross-over point

Chemical

Physical

Soybean

Palm

%FFA

0 0.5 1 1.5 2 2.5 3 3.5 4

3%
Deodorization conditions

Typical deodorization conditions

<table>
<thead>
<tr>
<th>Conditions</th>
<th>Chemical Refining</th>
<th>Physical</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>U.S.</td>
<td>Europe</td>
</tr>
<tr>
<td>Temperature (°C)</td>
<td>250-260</td>
<td>230-240</td>
</tr>
<tr>
<td>Pressure (mbar)</td>
<td>3-4</td>
<td>2-3</td>
</tr>
<tr>
<td>Sparge steam (%)</td>
<td>0.5-2.0</td>
<td>0.5-1.0</td>
</tr>
<tr>
<td>Time (min.)</td>
<td>20-40</td>
<td>40-60</td>
</tr>
<tr>
<td>Final FFA (%)</td>
<td>0.03-0.05</td>
<td>0.03-0.05</td>
</tr>
</tbody>
</table>

For Europe:
- Temperature (°C): 230-250
- Pressure (mbar): 2
- Sparge steam (%): 1-2
- Time (min.): 60-90
- Final FFA (%): 0.03-0.05
Deodorization principle

Stripping

FFA, volatile odoriferous components,
Valuable minor components (tocopherols, sterols, …)
Contaminants (pesticides, light PAH, PCB, dioxins, …)

Odor and taste removal (actual Deodorization)

Hydrolytic/thermolytic degradation : f (steam/ time)

Temperature effect

Heat bleaching, cis-trans isomerisation,
Polymerisation, interesterification,
Distillation-Determining Factors

- VOLATILITY of the components
  - Vapour pressure (at a given temperature)
  - General: heavier components are less volatile
  - FFA > Tocopherols > Sterols

- CONCENTRATION of the components
  - Partial pressure
  - Depends on vapour pressure and concentration
Refined Oil Quality

- Deodorization is a crucial refining stage
- Deodorizer design and process conditions have a determining effect on the refined oil quality

Control of 'unwanted' and 'desired' effects:
- trans fatty acid formation
- positional isomerisation of PUFA
- polymerisation (dimers)
- controlled stripping of tocopherols, sterols
- complete stripping of contaminants
Contaminant removal

• Adsorption on specific adsorbents (activated carbon)
  - Heavy polycyclic aromatic hydrocarbons
  - Dioxins and furans from Fish Oils
  - PCB (only partially, less efficient than dioxins)

• Deodorization (only 'volatile' contaminants)
  - Pesticides (organochlorine)
  - Light polycyclic aromatic hydrocarbons (coconut oil)
  - PCB, dioxins, brominated flame retardants (fish oil)
Deodorization Technology

Process stages

- Oil deaeration  ➔  Prevention oxidation
- Heating  ➔  Heat recovery
  Final heating
- Deodorization  ➔  Deacidification
  Injection of stripping steam
  Low pressure (vacuum)
  Condensation of volatiles
- Cooling  ➔  Heat recovery
  final cooling
- Polish filtration + AO dosing
Heating

Two stage process
- preheating followed by final heating

Preheating
- heat recovery step
- oil/oil heat exchanger (incoming oil/finished oil)

Final heating
- High pressure steam (most used & recommended today)
- Thermal oil (avoided for food safety reasons)
- Electrical heating (rarely used)
## Temperature of high pressure steam

<table>
<thead>
<tr>
<th>Pressure (bar)</th>
<th>Steam temperature (°C)</th>
<th>Latent heat (kJ/kg)</th>
<th>Specific volume (m³/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>99.6</td>
<td>2258</td>
<td>1.694</td>
</tr>
<tr>
<td>2</td>
<td>120.2</td>
<td>2202</td>
<td>0.8853</td>
</tr>
<tr>
<td>3</td>
<td>133.5</td>
<td>2163</td>
<td>0.6056</td>
</tr>
<tr>
<td>5</td>
<td>151.8</td>
<td>2108</td>
<td>0.3747</td>
</tr>
<tr>
<td>7</td>
<td>164.9</td>
<td>2065</td>
<td>0.2762</td>
</tr>
<tr>
<td>10</td>
<td>179.9</td>
<td>2014</td>
<td>0.1943</td>
</tr>
<tr>
<td>15</td>
<td>198.3</td>
<td>1945</td>
<td>0.1316</td>
</tr>
<tr>
<td>20</td>
<td>212.4</td>
<td>1889</td>
<td>0.09952</td>
</tr>
<tr>
<td>30</td>
<td>233.8</td>
<td>1794</td>
<td>0.06663</td>
</tr>
<tr>
<td>40</td>
<td>250.3</td>
<td>1713</td>
<td>0.04975</td>
</tr>
<tr>
<td>50</td>
<td>263.9</td>
<td>1640</td>
<td>0.03943</td>
</tr>
</tbody>
</table>
Heat recovery

External heat exchanger

- plate
- spiral
- shell & tube

Internal heat exchangers

- oil-steam heat exchanger
- oil-oil heat exchanger
Heat recovery: Thermosyphon

- L.P. Steam Heating
- H.P. Steam Heating
- L.P. Steam Generation

L.P. Steam Generation & Thermosyphon Heat Recovery 75%

Double Thermosyphon Heat Recovery 67%

Single Thermosyphon Heat Recovery 45%
Deodorizer design

Deep bed deodoriser

Shallow bed deodoriser
Vapor scrubbing system

Composition of vapor phase

- Volatile components (FFA, odor components)
- Stripping steam
- Non condensable gases (air,...)

Condensation of volatile components

- intimate contact between vapor and recirculating distillate
- series of sprayers or packed bed in vacuum duct
- Distillate is recirculating at the lowest possible temp.
- Installation of demister at the top
- Designed to have a minimal pressure drop
Vapor scrubbing system

Deaerator  Deodoriser  Vapor scrubber  Vacuum unit

heating  deodorising  cooling
## Deodorizer distillates

### Composition of industrial deodorizer distillates

<table>
<thead>
<tr>
<th>Component</th>
<th>Soybean chemical</th>
<th>Soybean physical</th>
<th>Corn chemical</th>
<th>Corn physical</th>
<th>Sunflower chemical</th>
<th>Sunflower physical</th>
</tr>
</thead>
<tbody>
<tr>
<td>Squalene (%)</td>
<td>1-2</td>
<td>0.5</td>
<td>0.5-1.0</td>
<td>0.5</td>
<td>0.5</td>
<td>0.5</td>
</tr>
<tr>
<td>Tocopherols (%)</td>
<td>16-20</td>
<td>5-7</td>
<td>2-4</td>
<td>5-7</td>
<td>1-2</td>
<td>1-2</td>
</tr>
<tr>
<td>Sterols (%)</td>
<td>19-23</td>
<td>11</td>
<td>3-6</td>
<td>12-14</td>
<td>4-5</td>
<td>4-5</td>
</tr>
<tr>
<td>Triglycerides (%)</td>
<td>5-6</td>
<td>4</td>
<td>1-2</td>
<td>2-3</td>
<td>1-2</td>
<td>1-2</td>
</tr>
<tr>
<td>FFA (%)</td>
<td>33</td>
<td>75</td>
<td>77-81</td>
<td>39</td>
<td>70</td>
<td></td>
</tr>
</tbody>
</table>

Concentration of contaminants (pesticides, PAH)
Vacuum systems

Conventional vacuum system

- Combination of steam jet ejectors (boosters), vapor condensers and mechanical (liquid-ring) vacuum pump

- High motive steam consumption (60-85% of total steam)

<table>
<thead>
<tr>
<th>Pressure</th>
<th>kg motive steam per kg stripping steam</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>30°C (1)</td>
</tr>
<tr>
<td>Booster</td>
<td>Deodorizer</td>
</tr>
<tr>
<td>2.5</td>
<td>3 mbar</td>
</tr>
<tr>
<td>1.5</td>
<td>2 mbar</td>
</tr>
</tbody>
</table>

Note: (1) Barometric condenser water inlet temperature: 24°C; outlet temperature: 30°C
(2) Barometric condenser water inlet temperature: 5°C; outlet temperature: 10°C;
Vacuum systems

- A process vapour from column
- B scrubber
- C to ejector vacuum system
- D hotwell
- E plate heat exchanger
- F cooling tower

1-3 boosters / ejector
4 stand by ejector
5-6 direct contact condensers
7 liquid ring vacuum pump
8 separator
Dry condensing - Ice condensing

- Sublimation of steam (into ice) on surface condensers
- Low pressure can be reached (< 2 mbar in deodorizer)
- Strongly reduced odor emission
- Nearly no motive steam but higher electricity consumption
- Higher investment cost (compared to boosters)
- Operating cost (and ROI) will depend on ratio between cost of steam and electricity

Generally shorter ROI in Europe
Dry condensing vacuum system with horizontal condensers
Dry Condensation Systems with vertical condensers

- From FA scrubber
- Condenser
- Separator
- Cooling water
- Vapour (vacuum)
- Refrigerant (ammonia)
- Non-condensable gases
- Valve, open
- Valve, closed

- Freeze condenser
- To de-aeration
- LP steam
- Condensate
- Melt vessel

- process vapor
- water
- ammonia
Deodorizer design

Batch deodorization

Continuous deodorization
- Horizontal deodorizer
- Single vessel vertical deodorizer
- Packed column technology

Semi-continuous deodorization
Continuous Deodorization

Limited feedstock changes

Advantages
- Low utilities cost (high heat recovery)
- Short residence time
- Excellent control of all parameters

Disadvantage
- Contamination during feedstock change
Continuous Deodorization

Continuous deodorizer types

- Horizontal multi-vessel deodorizer

- Vertical deodorizer → most common
  → all operations integrated in single vessel

- Thin film deodorizer → packed column
  + retention vessel
Continuous horizontal multi-vessel deodoriser
Packed column stripper

Liquid phase (oil)

Gas phase

To FAD scrubber + vacuum unit

Structured packing

Vapor phase (steam)

\[ F = V_{vap} \sqrt{\rho} \cdot A \]

ΔP : 0.1 - 0.5 mbar/m
ΔT : min. 1.3°C / %FFA
H : 3-5 m
D : f (vapor load)

To deodorizer
PACKED COLUMN TECHNOLOGY

• Specific Process conditions
  - Structured packing : 100 - 300 m²/m³
  - Efficient stripping : Counter-current contact oil/steam
  - Short residence time : Few minutes at high temperature

• Applications
  - Stripping of valuable minor components or contaminants from heat sensitive oils
  - Preferably only in continuous operation
  - No deodoriser (too short residence time)