Physical Refining

Feedstock Parameters

- Seed Oil (Soybean, Rapeseed, Sunflower)
  - FFA \( \leq 2\% \)
    - higher FFA indicates low quality oil and may not be suitable for physical refining
  - Phosphorous \( \leq 5 \text{ ppm}, \leq 2 \text{ desired} \)
  - Iron \( \leq 0.2 \text{ ppm} \)
Chemical Refining

Feedstock Parameters

- Seed Oil (Soybean, Rapeseed, Sunflower)
  - FFA \( \leq 3\% \)
  - Phosphorous \( \leq 1200 \text{ ppm}, \leq 200 \text{ ppm desired} \)
Purpose of Degumming

• Commercial Lecithin production
• Prevent crude oil settling during storage or transport
• Waste water (prevent acidulation of gums)
• Physical Refining
• Reduction in neutralisation losses
Gums

- Two main types
  - Hydratable Phosphatides - easy to remove
  - Non-Hydratable Phosphatides (NHP) - hard to remove from oil
    - Some NHP removed with hydratables in water degumming
    - requires the use of an acid to convert to hydratable for complete removal
## Gum content of various oils

<table>
<thead>
<tr>
<th>Oil type</th>
<th>Phosphatides (%)</th>
<th>Phosphorus (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coconut</td>
<td>0.02 – 0.05</td>
<td>10 – 20</td>
</tr>
<tr>
<td>Corn</td>
<td>0.7 – 2.0</td>
<td>250 – 800</td>
</tr>
<tr>
<td>Cottonseed</td>
<td>1.0 – 2.5</td>
<td>400 – 1000</td>
</tr>
<tr>
<td>Groundnut</td>
<td>0.3 – 0.7</td>
<td>100 – 300</td>
</tr>
<tr>
<td>Palm</td>
<td>0.03 – 0.1</td>
<td>15 – 30</td>
</tr>
<tr>
<td>Rapeseed</td>
<td>0.5 – 3.5</td>
<td>200 – 1400</td>
</tr>
<tr>
<td>Soya</td>
<td>1.0 – 3.0</td>
<td>400 – 1200</td>
</tr>
<tr>
<td>Sunflower</td>
<td>0.5 – 1.3</td>
<td>200 – 500</td>
</tr>
</tbody>
</table>
Water Degumming Process Steps

• Heat oil to 60 - 70 °C
• Water addition and mixing
• Hydration mixing 30 minutes
• Centrifugal separation of hydrated gums
• Vacuum drying of degummed oil
• Gums - dried for edible lecithin or recombined in meal
Water Degumming

Water → Heater → Steam → Mixer → Reactor → Separator → Gums → Vacuum Dryer → To storage

Heater

Steam

Vacuum dryer

To storage
Water Degumming

Target Results

• Phosphorous in oil - 50 to 200 ppm max.
• Al% in dried gums - 65 to 70%
• Moisture in dried oil - < 0.1%
Acid Degumming Process Steps

- Heat oil to 60 - 70 °C
- Acid addition and mixing
- Hydration mixing 30 minutes
- Centrifugal separation of hydrated gums
- Vacuum drying of degummed oil
- Gums - recombined in meal
Acid Degumming
Acid Degumming

Target Results

• Phosphorous in oil - 20 to 50 ppm max.
• Al% in dried gums - 65 to 70%
• Moisture in dried oil - < 0.1%
Major Deep Degumming Methods

- Alfa Laval Special Degumming
- Super/Uni Degumming
- TOP Degumming
- Organic Refining Process
- Soft Degumming
- Enzymatic Degumming
Deep Degumming

• Deep degumming utilizes a reagent like acid to chelate Iron, Calcium, and Magnesium away from the NHP complex. Once the Iron, Calcium, and Magnesium are removed from the NHP complex the phosphatide becomes hydratable.

• Enzymatic degumming utilizes an enzyme to modify the NHP into a hydratable form.
Alfa Laval Special Degumming

- Heat oil to 60 °C
- 0.05-0.2 % Phosphoric Acid with intensive mixing
- Partially neutralise with dilute lye (hydration water)
- Gentle mixing and holding for 60 minutes
- Gums centrifugation
- Optional water wash step for lower phosphorous
- Oil drying
Alfa Laval Special Degumming

- Crude oil
- Heater
- Mixer
- Acid
- Lye
- Water
- Reactor
- Oil temperature trimmer
- Cooling water
- Separator
- Steam
- Gums
- To drying/storage

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Alfa Laval Special Degumming

Target Results

- Phosphorous in oil - 20 to 30 ppm max.
- Phosphorous in oil - 8 to 10 ppm max. with washing
- AI% in dried gums - 50 to 60%
- Moisture in dried oil - < 0.1%
## Deep Degumming Results

<table>
<thead>
<tr>
<th>Process</th>
<th>Phosphatides (%)</th>
<th>Phosphorus (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Special Degumming</td>
<td>&lt; 0.02</td>
<td>&lt; 10</td>
</tr>
<tr>
<td>Super/Uni Degumming</td>
<td>0.01 – 0.04</td>
<td>5 – 15</td>
</tr>
<tr>
<td>TOP Degumming</td>
<td>0.01 – 0.02</td>
<td>5 – 10</td>
</tr>
<tr>
<td>Soft Degumming</td>
<td>&lt; 0.01</td>
<td>&lt; 5</td>
</tr>
<tr>
<td>ORP</td>
<td>&lt; 0.02</td>
<td>&lt; 10</td>
</tr>
<tr>
<td>Enzymatic Degumming</td>
<td>0.01 – 0.02</td>
<td>5 – 10</td>
</tr>
<tr>
<td>Ultrafiltration</td>
<td>&lt; 0.01</td>
<td>&lt; 5</td>
</tr>
</tbody>
</table>
Purpose of Alkali Refining

• Removing of impurities from oil
  – Phospholipids (gums)
  – Colour bodies
  – Metal Ions - Pro-oxidants
    • Iron
    • Copper
  – Free Fatty Acids
  – Solids - meal fines
Oil Impurities

• Phospholipids
  – cause emulsions
  – darken oil with heat
  – interfere with crystallization

• Colour Bodies
  – Some have nutritional value
  – Remove to add consumer appeal & functionality in industrial uses
    (not heat stable)

• Metal Ions
  – act as pro-oxidants degrading the oil quality & stability
Alkali Refining Process Steps

- **Crude Oil**
  - Acid Mixing
  - Lye Mixing
  - Heating
  - Separation
  - Soapstock to waste treatment

- **H$_3$PO$_4$**
  - Acid Mixing

- **NaOH**
  - Lye Mixing
  - Heating
  - Separation

- **Heating**
  - Water Addition
  - Separation
  - Vacuum Drying
  - Refined & Dried Oil

- **Hot Water**
Alkali Refining Plant - ca. 1892
Neutralization Reaction

\[ R - \overset{\text{II}}{\text{C}} - \text{OH} + \text{NaOH} \rightarrow R - \overset{\text{II}}{\text{C}} - \text{O-Na} + \text{H}_2\text{O} \]

Fatty Acid + Caustic Soda → Soap

Other mechanisms

- colour bodies adsorbed onto soap
- phosphatides hydrated by water in lye
- chelated metal ions removed in soap
Effect of Lye Excess

- Groundnut oil with 2.5% FFA refined with 4.25 N lye

<table>
<thead>
<tr>
<th>Lye excess (%)</th>
<th>FFASS (%)</th>
<th>Refining factor (excl. saponification)</th>
<th>Real refining factor (incl. saponification)</th>
</tr>
</thead>
<tbody>
<tr>
<td>5</td>
<td>55</td>
<td>1.82</td>
<td>1.86</td>
</tr>
<tr>
<td>40</td>
<td>75</td>
<td>1.33</td>
<td>1.61</td>
</tr>
<tr>
<td>80</td>
<td>80</td>
<td>1.25</td>
<td>1.81</td>
</tr>
</tbody>
</table>
## Effect of Acid Conditioning

<table>
<thead>
<tr>
<th>Oil</th>
<th>Amount of acid % w/w</th>
<th>Soap content (ppm)</th>
<th>P – content (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>Neutralised oil</td>
<td>Washed oil</td>
</tr>
<tr>
<td>Rapeseed</td>
<td>0</td>
<td>1900</td>
<td>1200</td>
</tr>
<tr>
<td></td>
<td>0.15</td>
<td>1500</td>
<td>80</td>
</tr>
<tr>
<td>Sunflower</td>
<td>0</td>
<td>1600</td>
<td>500</td>
</tr>
<tr>
<td></td>
<td>0.05</td>
<td>1400</td>
<td>50</td>
</tr>
<tr>
<td>Corn</td>
<td>0</td>
<td>2100</td>
<td>1500</td>
</tr>
<tr>
<td></td>
<td>0.10</td>
<td>1100</td>
<td>80</td>
</tr>
<tr>
<td>Soybean</td>
<td>0</td>
<td>700</td>
<td>120</td>
</tr>
<tr>
<td></td>
<td>0.10</td>
<td>600</td>
<td>40</td>
</tr>
</tbody>
</table>
Washing and Drying

• Washing
  - Hot soft process water should be used
  - 5-10% of oil flow

• Drying
  - 70 mm Hg vacuum
### Refining Process - Selection

<table>
<thead>
<tr>
<th>FFA</th>
<th>P - content</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>&lt; 200 ppm</td>
</tr>
<tr>
<td>&lt; 1,5 %</td>
<td>Long-Mix or Multi-Mix</td>
</tr>
<tr>
<td>1,5 – 3 %</td>
<td>Multi-Mix (2-stage)</td>
</tr>
<tr>
<td>&gt; 3 %</td>
<td></td>
</tr>
</tbody>
</table>
Long-Mix Process Steps

- Acid pre-treatment
- Lye mixing
- Retention mixing
- Emulsion break heating
- Centrifugal separation of soapstock
- Heat
- Water addition and mixing
- Centrifugal separation of wash water
- Vacuum drying of refined and washed oil
Long-Mix Process

Neutralization

Crude oil

Mixer

Retention mixers

Steam

Heater

Washing

Water

Steam

Heater

Separator

Soap-stock

Mixer

To drying/bleaching or storage

Acid

Lye
Long-Mix – Lye Treat Conditions

<table>
<thead>
<tr>
<th></th>
<th>Strength (°Bé)</th>
<th>Excess (%)</th>
<th>Reaction time (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Crude seed oils</td>
<td>14 - 22</td>
<td>70 - 100</td>
<td>3 – 6</td>
</tr>
<tr>
<td>Degummed seed oils</td>
<td>16 - 26</td>
<td>35 - 70</td>
<td>2 – 5</td>
</tr>
<tr>
<td>Cottonseed</td>
<td>18 - 36</td>
<td>70 - 200</td>
<td>6 – 10</td>
</tr>
<tr>
<td>Corn</td>
<td>18 - 20</td>
<td>35 - 100</td>
<td>1 - 2</td>
</tr>
</tbody>
</table>
Long-Mix

Target Results

- Phosphorous in oil after S1 - 8 to 12 ppm, 20 ppm max.
- Soap in oil after S1 - 200 to 300 ppm, 400 ppm max.
- Phosphorus in oil after S2 – max. 4 ppm
- Soap in oil after S2 – max. 50 ppm
- FFA in oil – 0,02 to 0,04 %, 0,05 % max.
- Moisture in oil – max. 0,5 % or 0,05% if dried
- Loss = max. 0,8 + 1,25 x TL
Multi-Mix Process Steps

- Heat
- Acid pre-treatment
- Lye mixing
- Centrifugal separation of soapstock
- Lye or water mixing
- Centrifugal separator of soapstock or wash water
- Water addition and mixing
- Centrifugal separation of wash water
- Vacuum drying of refined and washed oil
Multi-Mix Process

- Acid conditioning
  - Mixer
  - Neutralisation
  - Mixer
  - Rerefining or washing
  - Washing
  - Neutralised oil to drying/storage or bleaching

- Crude oil
  - Heater
  - Steam
- Acid conditioning
  - Separator
  - Soap-stock
  - Mixer
  - Make-up water
  - Process water
- Neutralised oil
  - Spent water
## Multi-Mix – Lye Treat Conditions

<table>
<thead>
<tr>
<th></th>
<th>Strength (°Bé)</th>
<th>Excess (%)</th>
<th>Reaction time (sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Crude seed oils</td>
<td>20 - 28</td>
<td>30 - 60</td>
<td>15 - 30</td>
</tr>
<tr>
<td>Degummed seed oils</td>
<td>20 - 28</td>
<td>10 - 30</td>
<td>1 - 3</td>
</tr>
<tr>
<td>Cottonseed</td>
<td>20 - 28</td>
<td>10 - 50</td>
<td>&gt;1</td>
</tr>
<tr>
<td>Anaimal &amp; Fish</td>
<td>20 - 28</td>
<td>10 - 30</td>
<td>1 - 3</td>
</tr>
</tbody>
</table>
Multi-Mix

Target Results

• Phosphorous in oil after S1 - 8 to 12 ppm, 20 ppm max.
• Soap in oil after S1 - 500 to 700 ppm, 1000 ppm max.

• Phosphorus in oil after S3 – max. 4 ppm
• Soap in oil after S3 – max. 30 ppm
• FFA in oil – 0,02 to 0,04 %, 0,05 % max.
• Moisture in oil – max. 0,5 % or 0,05% if dried
• Loss = max. 0,3 + 1,25 x TL (+ 0,3 % if re-refining)
Multi-Wax Process Steps

- Heat
- Acid pre-treatment
- Lye mixing
- Centrifugal separation of soapstock
- Lye mixing
- Cool
- Crystallization
- Heat
- Centrifugal separator of waxes and soapy water
- Heat
- Water addition and mixing
- Centrifugal separation of wash water
- Vacuum drying of refined and washed oil

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Multi-Wax

Target Results

• Soap in oil after S1 - 1500 to 2000 ppm, 2500 ppm max.

• Phosphorus in oil after S3 – max. 4 ppm

• Soap in oil after S3 – max. 50 ppm

• Wax removal – min. 85 %

• FFA in oil – 0,02 to 0,04 %, 0,05 % max.

• Moisture in oil – max. 0,5 % or 0,05% if dried

• Loss = max. 0,7 + 1,25 x TL for 600 ppm wax content or max. 1,2 + 1,25 x TL for 1500 ppm wax content
Other Refining Process

• Cold neutralization
  - combined neutralization and dewaxing

• Miscella refining
  - neutralization in the miscella phase

• Modified Caustic Refining (MCR)
Dry Refined Oil Quality Targets

• Soap - < 30 ppm
• Phosphorous - < 2 ppm
• Iron - < 0.2 ppm
• Copper - < 0.01 ppm
• FFA - < 0.05%
• Moisture - < 0.05%