Fractionation Technology and Continuous Fractional Crystallization.

A general view of fractionation and Facts Figures & Future Applications of continuous fractional crystallization

Desmet Ballestra Group

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Presentation Overview

- Fractionation system
  - Different systems and Crystallizers
  - Separation systems
  - Examples of the fractionation of different products

- iConFrac™ Continuous fractionation:
  - Facts: illustrating the science at basis of the technological development
  - Figures: Real industrial feedback figures, observations from 24/7 operation experience
  - Future: Demonstration of current applications and possibilities
Fractionation technologies

Palm oil

Crystallisation

Separation: Filtration

Filter press

Melting tank

Buffertank

Feed tank
Fractionation technologies

- **Dry fractionation** (no additives)
  most versatile application: food/nonfood

- **Wet fractionation** (NaLS / MgS)
  - crystallisation: continuous
  - separation: centrifuge

  Older plants for oleo-chemical industry (oleic acid)

- **Solvent fractionation** (acetone/hexane)
  - crystallisation: continuous or batch
  - separation: mostly; vacuum belt filter

  Today used for specialty fats, ex. HPMF and Shea butter
  Still new installations are built/ serious lack of cocoa butter expected
Solvent Fractionation technology

Sheabutter SPMF → Hexane/Acetone → Vacuum pump → Vacuum belt filter → Olein miscella → Solvent recovery → Solvent refinery → Olein refinery
Dry Fractionation technologies

**DRY FRACTIONATION**
- Economic and green technology (environmental friendly): no solvent, no chemicals, no effluent, no contamination, no losses
- Single and multi-step operations possible.
- Applicable to big range of products
Principles of Dry Fractionation

CRYSTALLIZATION from the melted state

Fully melting: to destroy the « thermal memory » of the fat

Crystal initiation (SFC of the slurry: ~1%)

Crystal growth (SFC of the slurry: ~10-25 %)

Oil temperature

Water temperature

°C

Hours

Adapted cooling to achieve the best selectivity
Winterization versus Fractionation

Winterizing = Removing minor components (waxes, traces of sat glycerol)

Applied on:
- Sunflower oil, Corn germ oil, …
  - continuous (< 0.5% solids)
- Ricebran oil, Cottonseed oil
  - > 2% solids
- Fish Oil,
  - > 5% solids

Fractionation!
Continuous Winterizing technology

Flow meter

Winterised Oil

Oil

Filter aid

Cold water

Crystalliser

Maturator

Filter aid

Spent filter aid

Horizontal leaf filters

Spent filter aid

Vertical leaf filters

Warm water
Combined Winterizing/ Fractionation technology

- Oil
- Cold water
- Warm water
- Maturator
- Filter aid
- Crystalliser 1
- Crystalliser 2
- Crystalliser 3
- Spent filter aid
- Olein
- Stearin
- Membrane filter press
- Polish filtration
- Warm water
- Filter aid
- Combined Winterizing/ Fractionation technology
Batch Winterizing technology

Cotton seed oil: Dewaxing and fractionation in 1 step
Tank crystallisers working in batch
Horizontal Leaf filters, with heating

Conventional crystallisers

Heated Hor. Leaf Filter
Batch or Continuous Winterizing technology

More performant crystallizers
Membrane Press technology for higher yield

Mobulizer®
Membrane press filter
Types of Cooling curves for Batch Fractionation

The oil can be cooled according to:

A) Oil temperature dependent water profile: $T_{\text{water}} = f(T_{\text{oil}})$

With Conventional Crystallizers

B) Fixed water cooling profile: $T_{\text{oil}} = f(T_{\text{water}})$

High performant Crystallizers

Figure 5.9 Schematic representation of an oil temperature related and independent water cooling profile.
Conventional Crystallizers

Horizontal cooling coils

Double cooling coils

Circular agitator, shear on crystals!
Conventional Crystallizers Desmet

Tirtiaux
Vertical cooling fins

Desmet TX
Vertical cooling pipes

Circular agitator, shear on crystals!
Desmet Crystallizers

Concentric crystalliser

Contube crystalliser

Can be pressurized
Resist high viscosity

Sweeping agitator for high heat exchange, shear on crystals!
Desmet Tubular Crystallizers

- Used also for Solvent fractionation
- Used also for FA fractionation
- Can work with scraper and high viscosity
- Can be pressurized

Sweeping or scraping agitator, shear on crystals!
MoBulizer Design: “Moving Bundle Crystallizer”

- Stainless steel pipe bundle
- 6m²/ton cooling surface, uniformly distributed over total volume
- Cooling & Agitation
- Low energy consumption
- Low shear on crystals
- High heat exchange efficiency
- Up to 50 Ton Capacity
- Optimized for iConFrac
MoBulizer Design: “Moving Bundle Crystallizer”

MOVING COOLING BUNDLES
Pilot Mobuliser plant
Full Flexible Cooling curve
Static Crystallization

Palm kernel oil

Palm kernel oil

25 µm
Crystallizer design Statolizer®

« Static » crystallization

Cooling plates
PKO static crystallization with Statolizer®

Dynamic pre-crystallization

Filtration

Fluidization

Oil Transfer

Static Crystallisation

Cake release

PKO static crystallization with Statolizer®
PKO fractionation with Statolizer®

Filtration
Membrane filter Presses

30 bar membrane filter press
Chambers: 15-30 mm gap
Specialty fats

Mixed pack plate design for 6-30 bar

6-16 bar membrane filter press
Chambers: 25-50 mm gap
Commodity oils
Vacuum belt filter

Vacuum drum filter

Filtration rate dependent (limited filtration surface !!)
Separation, (superscreen)centrifuge or decantor

Nozzle centrifuge Patented by GEA - Westfalia
# Comparison Membrane press filtration vs. centrifugation vs. Vacuum filtration

<table>
<thead>
<tr>
<th>Filtration data</th>
<th>Press filtration (16 bar)</th>
<th>Centrifugation</th>
<th>Vacuum filtration (drum / belt)</th>
</tr>
</thead>
<tbody>
<tr>
<td>IV palm oil</td>
<td>52</td>
<td>52</td>
<td>52</td>
</tr>
<tr>
<td>IV olein</td>
<td>57</td>
<td>57</td>
<td>57</td>
</tr>
<tr>
<td>IV stearin</td>
<td>32</td>
<td>38</td>
<td>40</td>
</tr>
<tr>
<td>SFC slurry</td>
<td>12%</td>
<td>12%</td>
<td>12%</td>
</tr>
<tr>
<td>SFC cake</td>
<td>65%</td>
<td>/</td>
<td>41%</td>
</tr>
<tr>
<td>Yield olein</td>
<td>&gt;80%</td>
<td>74-75%</td>
<td>71%</td>
</tr>
</tbody>
</table>

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Filtration data:
- **IV palm oil**: 52
- **IV olein**: 57
- **IV stearin**: 32
- **SFC slurry**: 12%
- **SFC cake**: 65%
- **Yield olein**: >80%

Press filtration (16 bar):
- **IV palm oil**: 52
- **IV olein**: 57
- **IV stearin**: 32
- **SFC slurry**: 12%
- **SFC cake**: 65%
- **Yield olein**: >80%

Centrifugation:
- **IV palm oil**: 52
- **IV olein**: 57
- **IV stearin**: 38
- **SFC slurry**: 12%
- **SFC cake**: /
- **Yield olein**: 74-75%

Vacuum filtration (drum / belt):
- **IV palm oil**: 52
- **IV olein**: 57
- **IV stearin**: 40
- **SFC slurry**: 12%
- **SFC cake**: 41%
- **Yield olein**: 71%
# Effect of squeezing and cake thickness

## Commodity fractions

<table>
<thead>
<tr>
<th>Squeezing pressure</th>
<th>SFC (1) Cake</th>
<th>Yield Stearin</th>
<th>Yield Olein</th>
<th>IV Stearin</th>
<th>IV Olein</th>
</tr>
</thead>
<tbody>
<tr>
<td>50 mm(2)</td>
<td>%</td>
<td>%</td>
<td>%</td>
<td>%</td>
<td>%</td>
</tr>
<tr>
<td>6 bar</td>
<td>55,0</td>
<td>23,6</td>
<td>76,4</td>
<td>39,7</td>
<td>57,1</td>
</tr>
<tr>
<td>15 bar</td>
<td>61,0</td>
<td>20,0</td>
<td>80,0</td>
<td>36,6</td>
<td>57,1</td>
</tr>
<tr>
<td>30 bar</td>
<td>65,0</td>
<td>18,3</td>
<td>81,7</td>
<td>34,7</td>
<td>57,1</td>
</tr>
<tr>
<td>25 mm(2)</td>
<td>%</td>
<td>%</td>
<td>%</td>
<td>%</td>
<td>%</td>
</tr>
<tr>
<td>6 bar</td>
<td>60,0</td>
<td>20,6</td>
<td>79,4</td>
<td>36,8</td>
<td>57,2</td>
</tr>
<tr>
<td>15 bar</td>
<td>66,0</td>
<td>18,8</td>
<td>81,2</td>
<td>34,9</td>
<td>57,2</td>
</tr>
<tr>
<td>30 bar(3)</td>
<td>70,0</td>
<td>16,2</td>
<td>83,8</td>
<td>32,1</td>
<td>57,2</td>
</tr>
</tbody>
</table>

### Notes:

1. SFC measured by NMR directly after cake discharge
2. Chamber width
3. No empty chamber guaranteed

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**Desmet Ballestra**
Multiple step fractionation from Palmoil.

Hard PMF: base for cacao butter equivalent (CBE)

- **Stearin IV 29-38**
- **Super stea IV 10-22**
- **Soft stea IV ~ 40**
- **PPP Stea IV ~ 25**
- **Soft PMF IV ~ 45**
- **Topped SPMF IV ~ 47**
- **Hard PMF**
  - IV ~ 34
  - SFC* @30°C: ~ 43%
  - DAG < 3%
  - SFC* @35°C: ~ 2%
  - PPP < 2%
  - SFC* @40°C: ~ 0%
  - POP ~ 65-67%
  - IUPAC tempered

- **RBD Palm Oil IV ~ 52**
- **Frying oil**
- **Olein IV 56-63**
- **85% Oleina IV 56/Palmoil**
- **Super Olein IV ~ 64-65**
- **Mid Olein IV ~ 52-53**

Salad oil
## Multi-step fractionation of PO, Increased Cold Stab.

<table>
<thead>
<tr>
<th>Cloud point (°C)</th>
<th>IV</th>
<th>% Olein in Soybean Oil for cold test at 0°C : 5.5 h</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mettler</td>
<td></td>
<td></td>
</tr>
<tr>
<td>7.2</td>
<td>57 (Olein)</td>
<td></td>
</tr>
<tr>
<td>2.5</td>
<td>63 (Superolein)</td>
<td>10</td>
</tr>
<tr>
<td>1.3</td>
<td>65 (Superolein)</td>
<td>30</td>
</tr>
<tr>
<td>-4.5</td>
<td>71 (Topolein)</td>
<td>100 (Cold test : 24h min. at 0°C)</td>
</tr>
<tr>
<td>0</td>
<td>69-71 (Hydrid olein)</td>
<td></td>
</tr>
</tbody>
</table>

![Graph showing the relationship between Mettler Cloud Point and % Palm Fraction for different IV values of Olein, Superolein, and Topolein.](image)
Multi-step fractionation of beef tallow fat.

Beef Tallow
DP: 43.2°C; IV: 44.2

- Olein: O
  DP: 18.9°C
  Recycling

- Stearin: S
  DP: 49°C

- Super Hard Stearin: SS
  DP: 54°C

- Olein: SO
  DP: 45°C

- Second Stearin: OS
  DP: 26.7°C

- Super Olein: OO
  DP: 14°C

- Third Stearin: OOS
  DP: 18.1°C

- Top Olein: OOO
  CP Mettler: -3°C

45% Recycling
30% Recycling
70% Recycling

DP: Dropping point
Multi-step fractionation of beef tallow fat.

Hard fractions;  
Semi-liquid  
Shortenings;  
frying shortenings;  
Margarine;  
Salad oils ...
Multi-step fractionation of Anhydrous Milk Fat (AMF)

AMF : DROPPING POINTS BETWEEN 45°C and 6°C ...
Triple fractionation : the best solution for all qualities of AMF

AMF
DP : 34°C

Hard Stearin : S
DP : 45°C

Olein : O
DP : 21°C

Second (Soft) Stearin : OS
DP : 26°C

Super Olein : OO
DP : 13°C

Third Stearin : OSS
DP : 32°C

Olein : OSO
DP : 26°C

Stearin : OOS
DP : 21°C

Top Olein : OOO
DP : 6°C

DP: Dropping point
Multi-step fractionation of (AMF) Spreadable butter.

- **AMF**
- **Soft Butter**
- **Spreadable butter**

Very soft or cooking butter
Fractionation of Fish Oil, Product development

**FISH OIL**

<table>
<thead>
<tr>
<th>FAC (% by GC)</th>
<th>Tuna</th>
<th>Anchovy</th>
<th>Cod liver</th>
<th>Menhaden</th>
<th>Catfish</th>
</tr>
</thead>
<tbody>
<tr>
<td>C14:0</td>
<td>3.1</td>
<td>8.4</td>
<td>4.3</td>
<td>7.7</td>
<td>3.2</td>
</tr>
<tr>
<td>C16:0</td>
<td>18.4</td>
<td>18.5</td>
<td>10.9</td>
<td>20.7</td>
<td>25.5</td>
</tr>
<tr>
<td>C18:0</td>
<td>5.8</td>
<td>3.6</td>
<td>2.4</td>
<td>3.8</td>
<td>7.4</td>
</tr>
<tr>
<td>SFA</td>
<td>30.2</td>
<td>32.0</td>
<td>19.3</td>
<td>33.4</td>
<td>36.6</td>
</tr>
<tr>
<td>C16:1</td>
<td>4.4</td>
<td>9.0</td>
<td>6.8</td>
<td>9.2</td>
<td>1.6</td>
</tr>
<tr>
<td>C18:1</td>
<td>18.1</td>
<td>11.4</td>
<td>20.8</td>
<td>12.9</td>
<td>43.0</td>
</tr>
<tr>
<td>C22:1</td>
<td>-</td>
<td>-</td>
<td>10.0</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>MUFA</td>
<td>25.3</td>
<td>24.8</td>
<td>41.3</td>
<td>26.5</td>
<td>46.3</td>
</tr>
<tr>
<td>C18:2</td>
<td>3.0</td>
<td>1.8</td>
<td>1.4</td>
<td>1.2</td>
<td>14.2</td>
</tr>
<tr>
<td>C18:4</td>
<td>-</td>
<td>2.5</td>
<td>13.5</td>
<td>3.1</td>
<td>-</td>
</tr>
<tr>
<td>EPA (C20:5 n3)</td>
<td>6.3</td>
<td>17.4</td>
<td>9.0</td>
<td>12.6</td>
<td>0.1</td>
</tr>
<tr>
<td>DHA (C22:6 n3)</td>
<td>24.5</td>
<td>11.0</td>
<td>10.1</td>
<td>13.4</td>
<td>0.3</td>
</tr>
<tr>
<td>PUFA</td>
<td>41.9</td>
<td>38.4</td>
<td>38.1</td>
<td>37.6</td>
<td>16.7</td>
</tr>
<tr>
<td>Cloud point (°C)</td>
<td>3.7</td>
<td>12.8</td>
<td>11.6</td>
<td>6.4</td>
<td>18.1</td>
</tr>
</tbody>
</table>

Objective: to increase the cold stability

FAC = f (type of fish oil)

High ω-3 oils (EPA/DHA)
**Fractionation of Fish Oil, Product development**

**Winterization: Single-step**

**Target: colt test@0°C: ~5 hrs**

**REDUCTION OF SFA / LIMITED INCREASE IN EPA/DHA**

**NB TUNA OIL**
- IV: 192
- Cloud point: 3.7°C
- SFA: 30.2%
- EPA+DHA: 30.8%

**STEARIN**
- IV: 148
- SFA: 44.7%
- EPA+DHA: 23.2%

**OLEIN**
- IV: 200
- Cloud point: -3.5°C
- SFA: 26.9%
- EPA+DHA: 32.5%
- Cold test: 5.5 hrs@0°C

**Yield:** 82% at 15 bar (40 mm chambers)
Fractionation of Fish Oil, Product development

**NB ANCHOVY OIL**
- IV: 187
- Cloud point: 12.8°C
- SFA: 32.0%
- EPA+DHA: 28.4%

**Winterization: Single-step**
- Target: colt test@0°C: ~3 hrs

**REDUCTION OF SFA / LIMITED INCREASE IN EPA/DHA**

**STEARIN**
- IV: 151
- SFA: 47.5%
- EPA+DHA: 20.4%

**OLEIN**
- IV: 193
- Cloud point: -3.2°C
- SFA: 29.5%
- EPA+DHA: 29.4%
- Cold test: 3 hrs@0°C

Yield: 78 % at 6 bar*
(40 mm chambers)

*: max.
Fractionation of Fish Oil, Product development

**NB COD LIVER OIL**
- IV: 158
- Cloud point: 11.6°C
- SFA: 19.3%
- EPA+DHA: 19.1%

**Winterization: Single-step**
- Target: cold test@0°C: ~24 hrs

**REDUCTION OF SFA / LIMITED INCREASE IN EPA/DHA**

**STEARIN**
- IV: 125
- SFA: 27.6%
- EPA+DHA: 13.5%

**OLEIN**
- IV: 164
- Cloud point: -14.9°C
- SFA: 17.7%
- EPA+DHA: 20.2%
- Cold test: > 24 hrs@0°C

Yield: 85% at 6 bar (40 mm chambers)
Fractionation of Lard fat.

Lard oil: known as very difficult to give filterable crystals

The Mobuliser is the solution, and is working industrially
Results of Fractionation of Lard oil in Mobuliser:

<table>
<thead>
<tr>
<th></th>
<th>Lard</th>
<th>Olein</th>
<th>Stearin</th>
</tr>
</thead>
<tbody>
<tr>
<td>Yield %</td>
<td></td>
<td>62</td>
<td>38</td>
</tr>
<tr>
<td>Cloud Point °C</td>
<td></td>
<td>0</td>
<td></td>
</tr>
<tr>
<td>SFC %</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>0 °C</td>
<td>38.4</td>
<td>25.7</td>
<td>66.8</td>
</tr>
<tr>
<td>10</td>
<td>33.6</td>
<td>10.3</td>
<td>64.7</td>
</tr>
<tr>
<td>15</td>
<td>29</td>
<td>3.8</td>
<td>62.3</td>
</tr>
<tr>
<td>20</td>
<td>23.8</td>
<td>0.2</td>
<td>59.1</td>
</tr>
<tr>
<td>30</td>
<td>5.5</td>
<td></td>
<td>45</td>
</tr>
<tr>
<td>40</td>
<td>1.8</td>
<td></td>
<td>7.2</td>
</tr>
<tr>
<td>50</td>
<td>0</td>
<td></td>
<td>0.2</td>
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</tbody>
</table>
Single stage Palm kernel fractionation with Statolizer® technology

Crude Palm Kernel oil IV 18

Single stage

Statoliser 18°C

PK Olein IV ~ 25
60-64 %

PK Stearin IV ~7
36-40 %

CBS IV ~1

Full Hydrogenation

SFC-content

<table>
<thead>
<tr>
<th>°C</th>
<th>%</th>
</tr>
</thead>
<tbody>
<tr>
<td>20</td>
<td>95</td>
</tr>
<tr>
<td>25</td>
<td>90</td>
</tr>
<tr>
<td>30</td>
<td>50</td>
</tr>
<tr>
<td>35</td>
<td>5</td>
</tr>
</tbody>
</table>
Single stage Palm kernel fractionation with Statolizer® technology

Crude Palm Kernel oil IV ~ 18

Statoliser 22°C

PK Olein IV ~ 23

PK Stearin IV ~ 5,0

CBS

<table>
<thead>
<tr>
<th>SFC-content</th>
<th></th>
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<tbody>
<tr>
<td>°C</td>
<td>%</td>
</tr>
<tr>
<td>20</td>
<td>94</td>
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<tr>
<td>25</td>
<td>84</td>
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<td>30</td>
<td>56</td>
</tr>
<tr>
<td>35</td>
<td>1</td>
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</table>
Palm kernel fractionation with Statolizer® technology
Product evaluation

<table>
<thead>
<tr>
<th>Parameters</th>
<th>Single stage: Fully Hydrogenated stearin</th>
<th>Single stage: Stearin As such</th>
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<tbody>
<tr>
<td>IV</td>
<td>&lt; 1</td>
<td>4.8</td>
</tr>
<tr>
<td>FA (%)</td>
<td>2</td>
<td>2</td>
</tr>
<tr>
<td>C10:0</td>
<td>53</td>
<td>54</td>
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<tr>
<td>C12:0</td>
<td>21</td>
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<td>C14:0</td>
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<td>C16:0</td>
<td>10</td>
<td>1</td>
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<td>C18:0</td>
<td>-</td>
<td>4</td>
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<tr>
<td>SFC (%)</td>
<td>10°C: 99</td>
<td>97</td>
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<tr>
<td></td>
<td>20°C: 98</td>
<td>94</td>
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<td>25°C: 92</td>
<td>84</td>
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<td></td>
<td>30°C: 50</td>
<td>56</td>
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<tr>
<td></td>
<td>35°C: 5</td>
<td>1</td>
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</tbody>
</table>
Oleochemistry

Dry Fractionation of Tallow Fatty Acids

Tallow Fatty Acids

<table>
<thead>
<tr>
<th>Component</th>
<th>Percentage</th>
</tr>
</thead>
<tbody>
<tr>
<td>I.V.</td>
<td>= 54</td>
</tr>
<tr>
<td>C14 &amp; lights</td>
<td>= 2.5%</td>
</tr>
<tr>
<td>C16:0</td>
<td>= 26%</td>
</tr>
<tr>
<td>C18:0</td>
<td>= 15%</td>
</tr>
<tr>
<td>C18:1</td>
<td>= 42%</td>
</tr>
<tr>
<td>C18:2/3</td>
<td>= 5%</td>
</tr>
</tbody>
</table>

Dry Fractionation

43-50 %

Oleic Acid

- I.V. = 85 - 93
- C.P. = <8
- C16:0 < 6%
- C18:0 0.9-1.8%
- C18:1 69-73%
- C18:2 7-10%

50-57 %

Stearic Acid

- I.V. = 16 - 25
- C16:0-C18:0 = 75-80%
- C18:1 15-19%
- C18:2 0.5-1%

Tallow FA Dry Fractionation
Oleochemistry
Fractionation of Fatty Acids of PK (PKFA)

Typical example (depleted 16-18)

Depleted PKO Fatty Acids
- I.V. = 64.9
- C14 & lights = 1.4%
- C16:0 = 25.7%
- C18:0 = 7.5%
- C18:1 = 56.1%
- C18:2 = 8.6%
- C18:1 / C18:2 = 6.5

40-50 %

Dry Fractionation

Oleic Acid
- I.V. = 90 - 100
- C.P. = 8
- C16 & lights < 10%
- C18:0 < 2%
- C18:1 > 75%
- C18:2 12%

Stearic Acid
- I.V. = 30 - 45
- C16:0-C18:0 = 58%
- C18:1 = 38%
- C18:2 = 8%

50-60 %

PKFA Dry Fractionation

C16-C18
26.1 tons
Biodiesel
Fractionation Palm Oil Methyl Esters

RBD Palm Oil
IV ~52

TRANSESTERIFICATION

Palm Oil Methyl Ester
IV ~52 CP 11,1

FRACTIONATION

1
Olein of Methyl Esters
IV ~60
Mettler cloud point: 7.6°C
C16:0: 37.1%
Yield ~56%

2
Olein of Methyl Esters
IV ~67
Mettler cloud point: 5.0°C
C16:0: 30.5%
Yield ~37%

3
Olein of Methyl Esters
IV ~73
Mettler cloud point: 2.1°C
C16:0: 23.6%
Yield ~24%
Conclusions of this section of general fractionation

Adapt the optimum fractionation system to the oil or fat:

In practice, consideration for **CRYSTALLIZATION**; the key factors are:

1) **APPROPRIATE COOLING SURFACE**   Conducted under:
2) **GOOD HEAT/MASS TRANSFER**
3) **NO CRYSTAL FRAGMENTATION**
4) **HIGH SHEAR**
5) **LOW SHEAR**
6) **IN STATIC**

CONCEPTUAL CHOICES:

**BATCH CRYSTALLIZATION**

- **SLOW CRYSTALLIZATION (LONG CYCLE TIME)**; Conventional Crystallizers versus
- **QUICK CRYSTALLIZATION (SHORT CYCLE TIME)**; Desmet principle

**STATIC CRYSTALLIZATION**: Requested when dynamic fails

**CONTINUOUS CRYSTALLIZATION**; 1st choice for Lower Opex;
Low shear crystallizer request, Mobuliser
Continuous Fractional Crystallization Technology

Facts, Figures & Future Applications
Introducing iConFrac™ Technology:

- **Patented** technology with signature equipment
- **Continuous crystallization** in combination with semi-continuous high pressure membrane press filtration
- Widely adopted in the industry since its **global launch in 2011**
- Specifically targeting **palm oil** industry but spreading to other oils and fats.
Crystallization in Batch: “Reaction in Time”

Temperature: 60°C
Solid Fat Content: 0%
Viscosity: 50 cP

Temperature: 22°C
Solid Fat Content: 10%
Viscosity: 350 cP
iConFrac: “Reaction in Space”

**Temperature:** 40°C  
**Solid Fat Content:** 0%  
**Viscosity:** 60 cP

**Temperature:** 22°C  
**Solid Fat Content:** 10%  
**Viscosity:** 350 cP
Why the focus on Crystallization?

- Utilities consumption in filtration is insignificant compared to crystallization
- To reduce opex, focus is on crystallization and heat recuperation of fractions
FACTS: Design behind the Technology

1. Sustain Plug-Flow in MoBulizer
2. Improve Crystallization Selectivity
3. Patented Heat Flush System
Particle Traces Colored by Particle Residence Time (s)  (Time=3.7500e-01)  
Crank Angle=103.50(deg)  

Jul 08, 2014  
ANSYS Fluent 15.0 (2d, dp, pbns, dynamesh, ske, transient)
• **FACTS:** Science behind the Technology

1. Sustain Plug-Flow in MoBulizer

   - *Specific Baffle Geometry to favor horizontal mixing*
   - *Dynamic distribution over MoBulizer width*
   - *Density differences: oil vs. crystals & warm vs. cold*
How does it affect cold stability?

Batch ‘cooling path’
- When oil enters the crystallizer, a much higher DeltaT is realised
- Meanwhile, oil is continuously exposed to cold heat exchange surface

Continuous ‘cooling path’
Proof of Principle: Palm olein IV 56-57

Set-up:

- 16 barg Filtration

Result:

<table>
<thead>
<tr>
<th>Triglycerides in Olein</th>
<th>iConFrac</th>
<th>Batch</th>
</tr>
</thead>
<tbody>
<tr>
<td>DiGly</td>
<td>7.53</td>
<td>7.87</td>
</tr>
<tr>
<td>ECN44</td>
<td>3.37</td>
<td>3.26</td>
</tr>
<tr>
<td>OLO</td>
<td>1.85</td>
<td>1.84</td>
</tr>
<tr>
<td>POL</td>
<td>10.77</td>
<td>11.07</td>
</tr>
<tr>
<td>PLP</td>
<td>9.77</td>
<td>9.95</td>
</tr>
<tr>
<td>MPO</td>
<td>0.18</td>
<td>0.14</td>
</tr>
<tr>
<td>OOO</td>
<td>4.26</td>
<td>3.86</td>
</tr>
<tr>
<td>POO</td>
<td>24.77</td>
<td>23.92</td>
</tr>
<tr>
<td>POP</td>
<td>28.10</td>
<td>28.13</td>
</tr>
<tr>
<td>PPP</td>
<td>0.31</td>
<td>1.08</td>
</tr>
<tr>
<td>SOO</td>
<td>2.79</td>
<td>2.67</td>
</tr>
<tr>
<td>POS</td>
<td>5.30</td>
<td>4.92</td>
</tr>
<tr>
<td>SOS</td>
<td>0.72</td>
<td>0.75</td>
</tr>
<tr>
<td>PPS</td>
<td>-</td>
<td>0.19</td>
</tr>
</tbody>
</table>

POP/PPP: ~100 ~30
FACTS: Science behind the Technology

2. Improve Crystallization Selectivity
   - Different ‘Cooling pattern’ compared to batch
   - Results in small, highly filterable crystals
   - Higher selectivity leads to better cold stability
What about the crystal deposit?

3. Patented Heat Flush System

- Crystal deposits slowly encrust the heat exchange surface, reducing efficiency
- In iConFrac, deposits are automatically melted off at regular basis by a ‘shot’ of hot water
- But: Continuous production is NOT interrupted (no loss of capacity)

Melted deposits do not affect cloud point of olein

Example for production of Olein IV 56, the PPP-content in the daily olein tonnage increases by max 0.03%, PPP increase is negligible
• **FACTS:** Science behind the Technology

1. Sustain Plug-Flow in MoBulizer

2. Improve Crystallization Selectivity

3. Patented Heat Flush system
   - *Automatic, self-cleaning*
   - *No interruption of continuity*
   - *No negative effect on cloud point or cold stability*
- **FIGURES:** Industrial Results & Experience

1. Performances

2. Versatility of Operation

3. Ease of use
Overall Flowchart
Olein Yields for RBD Palm Oil

<table>
<thead>
<tr>
<th>Batch</th>
<th>Continuous</th>
</tr>
</thead>
<tbody>
<tr>
<td>56</td>
<td>+ 2%</td>
</tr>
<tr>
<td>57</td>
<td>+ 3%</td>
</tr>
<tr>
<td>58</td>
<td>+ 3%</td>
</tr>
<tr>
<td>60</td>
<td>+ 2%</td>
</tr>
<tr>
<td>62</td>
<td>+ 2%</td>
</tr>
</tbody>
</table>
Steam Consumption for RBD Palm Oil

<table>
<thead>
<tr>
<th>Batch Consumption (%)</th>
<th>Continuous Consumption (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>- 40 %</td>
<td>- 40 %</td>
</tr>
<tr>
<td>35 %</td>
<td>33 %</td>
</tr>
</tbody>
</table>

Steam consumed (kg/ton)
Electricity Consumption for RBD Palm Oil

<table>
<thead>
<tr>
<th>Batch</th>
<th>Continuous</th>
</tr>
</thead>
<tbody>
<tr>
<td>-25%</td>
<td>-30%</td>
</tr>
<tr>
<td>-30%</td>
<td>-30%</td>
</tr>
</tbody>
</table>

Electricity used (kWh/ton)
FIGURES: Industrial Results & Experience

1. Performances

- Constant filling and draining of crystallizer: Time gain
- Higher Yields & Lower OPEX
- Smaller peak loads for pumps, cooling, heating..
Flexibility?  (IV 56 production) 4 Mob up to 1100 TPD

Operating 4 Units Parallel
400 tpd flowchart (IV 62 production)

Operating 2x2 Units in series
For more delicate products
(superolein production)
200 tpd flowchart (IV 65 production)

Operating 4 Units in series

For more delicate products (or olein refractionation)
2. Versatility of Operation

- Palm oil, Superoleins, soft PMF,…
- Minimal start-up & shut-down time: batch-like flexibility
Ease of Operation: simple automation

<table>
<thead>
<tr>
<th>STEP</th>
<th>Temp H2O °C</th>
<th>Min H2O Temp</th>
<th>Temp OIL °C</th>
<th>DELTA T °C</th>
<th>RAMP min.</th>
<th>ISO time min.</th>
<th>STEP min.</th>
<th>VELOCITY rpm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 O</td>
<td>+60.0</td>
<td>+55.0</td>
<td>+62.0</td>
<td>+5.0</td>
<td>+10</td>
<td>+5</td>
<td>+5</td>
<td>+8</td>
</tr>
<tr>
<td>2 W</td>
<td>+50.0</td>
<td>+45.0</td>
<td>+52.0</td>
<td>+5.0</td>
<td>+20</td>
<td>+10</td>
<td>+30</td>
<td>+8</td>
</tr>
<tr>
<td>3 W</td>
<td>+45.0</td>
<td>+40.0</td>
<td>+47.0</td>
<td>+5.0</td>
<td>+10</td>
<td>+5</td>
<td>+15</td>
<td>+8</td>
</tr>
<tr>
<td>4 W</td>
<td>+40.0</td>
<td>+35.0</td>
<td>+42.0</td>
<td>+5.0</td>
<td>+20</td>
<td>+10</td>
<td>+30</td>
<td>+8</td>
</tr>
<tr>
<td>5 D</td>
<td>+35.0</td>
<td>+30.0</td>
<td>+37.0</td>
<td>+4.0</td>
<td>+10</td>
<td>+5</td>
<td>+5</td>
<td>+6</td>
</tr>
<tr>
<td>6 D</td>
<td>+30.0</td>
<td>+25.0</td>
<td>+32.0</td>
<td>+4.0</td>
<td>+10</td>
<td>+10</td>
<td>+10</td>
<td>+6</td>
</tr>
<tr>
<td>7 D</td>
<td>+25.0</td>
<td>+20.0</td>
<td>+27.0</td>
<td>+4.0</td>
<td>+10</td>
<td>+5</td>
<td>+5</td>
<td>+6</td>
</tr>
<tr>
<td>8 O</td>
<td>+20.0</td>
<td>+15.0</td>
<td>+22.0</td>
<td>+4.0</td>
<td>+10</td>
<td>+10</td>
<td>+10</td>
<td>+6</td>
</tr>
<tr>
<td>9 W</td>
<td>+15.0</td>
<td>+10.0</td>
<td>+17.0</td>
<td>+3.0</td>
<td>+10</td>
<td>+5</td>
<td>+15</td>
<td>+5</td>
</tr>
<tr>
<td>10 W</td>
<td>+15.0</td>
<td>+10.0</td>
<td>+17.0</td>
<td>+3.0</td>
<td>+20</td>
<td>+10</td>
<td>+30</td>
<td>+5</td>
</tr>
<tr>
<td>11 W</td>
<td>+14.0</td>
<td>+10.0</td>
<td>+16.0</td>
<td>+3.0</td>
<td>+10</td>
<td>+5</td>
<td>+15</td>
<td>+5</td>
</tr>
<tr>
<td>12 W</td>
<td>+14.0</td>
<td>+10.0</td>
<td>+16.0</td>
<td>+3.0</td>
<td>+20</td>
<td>+10</td>
<td>+30</td>
<td>+5</td>
</tr>
</tbody>
</table>

Oil Flowrate (tph) | 10
Water Temperature (°C) | 21
Agitation (rpm) | 6

iConFrac: only 3 process settings!
+ 2 settings per Mobulizer in series

Batch: can be over 50 process settings!
FIGURES: Industrial Results & Experience

1. Performances

2. Versatility of Operation

3. Ease of use

- Simple and transparent automation
- Generally achieving performance faster than batch plants!
- Current Record: 150 consecutive production days
And so, iConFrac was introduced in 2011:
iConFrac is a globally applied technology:

4 years later, more than 20 installations have been sold over 4 continents
iConFrac is already having repeat orders:

Repeat orders as testimony of industrial reliability and performance
Future Applications: Push things forward

New paths to better products

IV 66-67 via online tie-in, or superolein straight from Palm oil

Superstearin (IV 10-13) production in 1 passage
200 tpd flowchart (IV 65 production)

Operating 4 Units in series
For more delicate products (or olein refractionation)
Easily achieve IV 66 by online tie-in
Or IV 67 by online tie-in

Cold superolein

RBD Palm Oil

PF1001A2

Outgoing Palm Olein

TO FILTRATION

RBD Palm Oil

PF1002A1

PF1002A2

PF1002A3

PF1002A4

DN100

F1081B2

F1081B1

F1082A

MF1082A

PF1078D

PF1078C

PF1050/82A

Cold superolein
...or IV 70 by online multiple tie-in
Future Applications: Push things forward

1. Optimized MoBulizer design further
   Enhance distribution and Baffle Geometries (mechanical design)

2. New paths to better products
   IV 66-67 via online tie-in, or superolein straight from Palm oil
   Superstearin (IV 10-13) production in 1 passage

Current development to link up seamlessly with Hard PMF production

Hybrid “iConFrac – Statolizer”
Continuous Fractional Crystallization Technology was introduced to the public on PIPOC Conference 2011…

4 years later, we can say:

- **Facts**: entirely based on sound scientific principles, Science behind Technology
- **Figures**: Significantly improved performance with smooth operation
- **Future**: Expanded applications and proven possibilities, even as far as Speciality fats
Thank you for your attention!