Fat Modification Processes: 
Dry Fractionation, Chemical and Enzymatic Interesterification and Hydrogenation

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Presentation outline

1. What about fat modification?

2. Dry Fractionation

3. Chemical & Enzymatic Interesterification

4. Hydrogenation

5. Conclusions
Total Yearly Edible Oil Production: 169 Mio tons

- Oversupply LIQUID oils
- Oil Modification required for use in food

- Palm Oil (28%)
- Soybean Oil (27%)
- Rapeseed oil (14%)
- Others (24%)
- Sunflower Oil (7%)
- CNO + PKO (5%)
Oils & Fats Processing

Seed → Preparation
- Cleaning / Drying
- Cracking
- Dehulling
- Flaking

Extraction
- Mechanical Extraction
- Solvent Extraction

Meal → Crude Oil

Modification
- Hydrogenation
- Interesterification
- Fractionation

Edible Oil

Refining
- Degumming
- Neutralising
- Bleaching
- Winterising
- Deodorising

Desmet Ballestra
EU margarine industry sets stricter trans fat standards

By Caroline Scott-Thomas, 19-Mar-2013

Related topics: Fats & oils, Legislation, Sugar, salt and fat reduction

The trade association representing margarine and vegetable fats makers in the EU has tightened its Code of Conduct for the third time since 1995, in an effort to reduce trans fat levels in foods and fats at retail.

The International Margarine Association of the Countries of Europe (IMACE) says that voluntary measures to reduce trans fat in margarines sold to food manufacturers already have led to an average 76% reduction since 2004, from 7.1% to 1.7%. The organisation also supports mandatory labelling of trans fatty acids from all sources for retail products.
Oils & Fats – New quality criteria

**OIL QUALITY**

**“STANDARDS”**

- Organoleptic properties/oxidative stability
  - Bland taste, odourless
  - Light and brilliant colour
  - Good oxidation parameters (PV and An-V)
  - Good shelf life
  - Good cold stability

**“NEW”**

**HEALTH CONCERNS**

**Nutritional qualities**
- Balanced FA composition
- High vitamin content (tocos ...)
- Low or zero *trans* FA
- No contaminants
- No fat degradation products

**ENVIRONMENTAL ISSUES**
- No effluent; no waste
- No chemicals; no solvent
- Low risk
- Lower oil losses
**Trans-Fatty Acids**

- Double bond naturally occurring in *cis* configuration
- *cis-trans* isomerization occurs mainly during partial hydrogenation
- ‘Natural’ *trans* fatty acids are formed in the rumen of ruminants and can be found in milk fats and animal fats
Trans-Fatty Acids: General characteristics

* Geometrical structure comparable to Saturated Fatty acids

* Trans isomers have higher melting point
  C18:1cis (oleic acid): 14°C
  C18:1trans (elaidic acid): 43.7°C

* Generally unwanted in food fats because of negative nutritional properties

* Sometimes desired in confectionery fats (CBR) for typical melting characteristics
Trans-Fatty Acids: Nutritional aspects

- Convincing evidence that consumption of trans FA gives:
  - increase of total/LDL cholesterol in blood (dose dependent)
  - increased risk for coronary heart disease

- Ruminant (natural) versus Industrial trans FA
  - No evidence that nutritional properties are different

- Dietary recommendations
  - As low as possible (EFSA)
  - < 1% Energy equals approx. max. 2 g trans FA/day (FAO)
Low *trans* Food Fats

Regulatory situation

- Max. *trans* levels in Food (fats) are set in various countries
- In US, Preliminary determination of FDA: *trans* FA are not GRAS

*Trans* Fatty Acids are banned from Food Fats

Partial hydrogenation replaced by dry fractionation and (chemical/enzymatic) interesterification
Oils & Fats – Why Modification?

Most edible oils have limited applications as functional ingredient in food products in the original form.

Modification technologies are applied to extend and increase their use in food formulation.

This is achieved by physical or chemical modifications of the triacylglycerol composition.

Historically, this was mostly done by partial, selective hydrogenation.

Nowadays, hydrogenated products are more and more replaced by low or non trans products.

The alternative: Fractionation & interesterification technologies!!
Oil Modification Technologies

INTERESTERIFICATION
- Chemical or Enzymatic process
- No change of FA profile
- Random triglyceride composition

HYDROGENATION
- Chemical process
- Saturation/isomerization
- Formation of trans FA

DRY FRACTIONATION
- Physical and Fully Reversible
- No solvents, No chemicals
- Yielding (min.) 2 fractions with different TAG compos.
Outline Presentation

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Dry Fractionation

Principle

• Controlled cooling of a melted fat in a special designed crystallizer
• Separation of crystals and liquid oil (filtration or centrifugation)
• Gives two fractions: Solid (‘Stearin’) and Liquid (‘Olein’)

Process characteristics

• Physical and fully reversible process
• No solvents, no additives, no chemicals: ‘Green Process’
• No yield loss, no post-treatment required
Dry Fractionation: Applications

**Food**

- **OIL (TAG)**
  - Stearin
  - Olein

**Oleochemistry**

- **FATTY ACIDS (FA)**
  - « stearic » acid
  - « oleic » acid

**Biofuel**

- **METHYL ESTERS (FAME)**
  - « stearic » FAME
  - « oleic » FAME

To improve cold stability = winterization

To improve physical properties
Dry Fractionation Process

Physical separation of triglycerides

Sat-Sat-Sat  Sat-Unsat-Sat  Sat-Unsat-Unsat  Unsat-Unsat-Unsat
SSS           SUS            SUU             UUU

Crystallization + Separation

Feedstock

Sat-Sat-Sat  Sat-Unsat-Sat
SSS           SUS

STEARIN

- Good physical properties
- Use in margarines or as shortening

Sat-Unsat-Unsat  Unsat-Unsat-Unsat
SUU             UUU

OLEIN

- Good cold stability
- Use as salad oil or frying oil
Dry Fractionation Process

<table>
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<th>Temperature (°C)</th>
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<th>Viscosity</th>
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**OIL**

**SSS / SSU**

**SUU / UUU**

**Crystallisation**

**Filtration**

**Solid Stearin**

**Liquid Olein**

**Palm Oil Fractionation Cooling Curve**

- rpm
- Oil temp
- Viscosity
- Water
- Solid Fat Content
Dry Fractionation: Different Crystallizers

High Shear Crystallizers: (1) Tirtiaux type

Static cooling Bundles or Fins

Agitation Blades
Dry Fractionation: Different Crystallizers

High Shear Crystallizers

(2) Concentric crystallizer

(3) Tubular crystallizer
Dry Fractionation : Different Crystallizers

Low Shear Crystallizer : MOBULIZER

MOVING COOLING BUNDLES
MOBULIZER = Moving Bundle Crystallizer

- Crystallizer with integrated cooling-agitation,
- Shear on crystals is minimized, but homogenous cooling with low speed Moving Bundle covering total volume
STATOLIZER = ‘STATIC’ Crystallizer

Static Crystallisation

Dynamic pre-crystallisation

Cake release

Cake Fluidisation

Oil Transfer

Filtration
Dry Fractionation: Separation of fractions

1. Vacuum Filters: Rotary drum or belt
   * Continuous filtration

2. Centrifugal Separation
   * Nozzle centrifuges or centrifugal decanters

3. Membrane press filters
   * Most applied filters in dry fractionation
   * Highest olein yield and best stearin quality
   * Discontinuous filtration (filtration cycle)
Dry Fractionation: Membrane Press Filter

Discontinuous filtration

1. Filling (2-3 bar)

2. Squeezing (6-30 bar)

3. Filter opening

Olein collection

Stearin collection
Dry Fractionation: Membrane Press Filter

- Membrane plate
- Filter plate
- Filter cloth
- Inflatable membrane
- Chamber (10-50 mm width) filled with crystallized oil
Dry Fractionation: new developments

Continuous crystallization = \text{iConFrac}^\text{™}
Crystallization in Batch: “Reaction in Time”

**SECTION A-A**

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

**SECTION A-A**

- **Temperature:** 22°C
- **Solid Fat Content:** 10%
- **Viscosity:** 350 cP
Continuous Crystallization: “Reaction in Space”

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

Achieved with MOBULIZER

- Gentle, laminar mixing
- Good heat transfer

- Internal baffles for better horizontal mixing

Temperature: 22°C
Solid Fat Content: 10%
Viscosity: 350 cP
1000 tpd flowchart (Palm Olein - IV 56 production)

Operating 4 Units Parallel
500 tpd flowchart (Palm Olein IV 58-60 production)

Operating 2x2 Units in series
For more delicate products (superolein production)
250 tpd flowchart (Palm Olein - IV 62 production)

Operating
4 Units in series
For more delicate products
(or olein refractionation)
Process Performance- Palm Olein IV 56-58

- Up to 15% higher throughput
- Up to 2% netto increased yield
- 45% less steam
- 35% less electricity
- 25% better filtration

iConFrac™

Batch
Continuous
iConFrac is a Proven Industrial Technology

- **2005-2010**: Pilot tests
- **2011**: First results presented at PIPOC
- **2012**: New Sales in **Malaysia, Africa, South America** 3500 tpd
- **2013**: New Sales in **Indonesia, India, South America** 4000 tpd

**CFD simulations**

**Start-up First Industrial Plant**
- **Europe** 750 tpd

**Start-up Second Industrial Plant**
- **Indonesia** 500 tpd
Dry Fractionation – Product development

Multi-stage Fractionation of Refined Palm Oil

New developments:
1. Refractionation of palm stearin
2. Refractionation of super olein
3. Production of good quality H-PMF

Diagram:
- RBD Palm Oil
  - Stearin: IV 32-36
  - Olein: IV 52-58
    - Soft PMF: IV 42-48
    - Hard PMF: IV 32-36
    - Mid olein: IV 52-53
    - Super olein: IV 64-66
    - Salad oil
Palm Oil Fractionation – Product development

1. Palm stearin refractionation

- **RBD Palm Oil**
  - IV ~ 52
  - PPP ~ 5% & POP ~ 28%

- **Olein**
  - IV ~ 56

- **Stearin**
  - IV ~ 33-34
  - PPP ~ 25% & POP ~ 28%

- **Super Stearin**
  - IV ~ 12-14
  - PPP ~ 65%
  - POP ~ 10%
  - SFC@10°C: ~ 95%
  - SFC@40°C: ~ 85%
  - SFC@60°C: ~ 20%

- **Olein**
  - IV ~ 56

- **Soft Stearin**
  - IV ~ 41-43
  - PPP ~ 10%
  - POP ~ 34%
  - SFC@10°C: ~ 75%
  - SFC@25°C: ~ 43%
  - SFC@40°C: ~ 16%

- **Frying oil**

- **Margarine and baking fats**

- **Dairy substitutes**

- **“Low trans” substitute of hydrogenated products**

- **Hardstock for margarines**
Palm Oil Fractionation – Product development

2. Super Olein refractionation

- **RBD Palm Oil**
  - IV ~ 52
  - ~ 20% Stearin IV 33-34
  - ~ 80% Olein IV ~ 56 CP: 10°C
  - 15 bar

- **Olein**
  - CP: 10°C
  - ~ 55%
  - ~ 55%
  - 15 bar
  - ~ 55%
  - ~ 55%

- **Soft PMF**
  - IV ~ 45
  - ~ 45%
  - 15 bar

- **Super olein**
  - IV ~ 64-65 CP: 4°C
  - ~ 50%
  - ~ 50%
  - < 15 bar

- **Top olein**
  - IV ~ 72-73 CP: -5°C
  - ~ 50%
  - ~ 50%

- **Margarine fat**
  - CP: 4°C

- **Recycling**
  - CP: -5°C
3 Production of good quality Hard PMF

- Traditionally done with solvent fractionation
- Challenge to produce good quality H-PMF via dry fractionation (fractionation route; crystallizer design, yield vs quality)
Palm Kernel Oil Fractionation

Single stage fractionation

Crude Palm Kernel oil
IV ~ 18

Statoliser
18°C

PK Olein
IV < 25

PK Stearin
IV = 7

CBS
IV ~ 1

Full Hydrogenation

Done with Static crystallizer
Palm Kernel Oil Fractionation

Two stage fractionation

Crude Palm Kernel oil
IV ~ 18

Statoliser
22°C

PK Olein
IV ~ 23

PK Stearin
IV ~ 5,0

CBS

SFC-content

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Direct use – No hydrogenation

To refractionation
Palm Kernel Oil Fractionation

Two stage fractionation: Olein refractionation

Crude Palm Kernel Olein
IV ~ 23

Statoliser
22°C

PK Olein
IV ~ 28

PK Stearin
IV ~ 7

CBS
IV ~ 1

Full Hydrogenation

81-83 %

17-19 %

Coating fats, filling creams
Basestock for interesterification

SFC-content

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Presentation outline

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Interesterification: Principle

- Rearrangement of FA (Random TAG composition)
- No change of the FA profile (no *trans* formation)
- One feedstock gives one interesterified oil
- Chemical catalyst or enzyme (irreversible)
Interesterification: Objectives

- Improve melting curve (steeper and/or lower melting point)
- Improve physical properties (e.g. plasticity)
- Combine properties of the oil blend

Palm Oil/Fully Hydro Palm Kernel Oil (60/40 w/w)

Palm Stearin/Coconut Oil (50/50 w/w)
Chemical Interesterification Process

- Feedstock: neutralized, bleached or fully refined

1. Drying + Interesterification
   * Drying at 120-150°C & 20 mbar to moisture < 100 ppm
   * Reaction: 30 min. at 90-120°C & 50 mbar with 0.05-0.1% NaOCH₃

2. Catalyst inactivation with water or citric acid solution

3. Bleaching (to remove color bodies and soaps)

4. Deodorization (FFA & FAME stripping, off-flavor removal)
Oil losses during Chemical Interesteriﬁcation

- Losses from catalyst addition
  * 1 mole NaOCH\(_3\) gives 1 mole soap and 1 mole FAME
  * Hence, 0.1% NaOCH\(_3\) gives 1% (soap + FAME)
  * Soap is converted in FFA during catalyst inactivation with citric acid
  * FFA and FAME are both removed during deodorization (stripping)

- Losses during bleaching & deodorization
  * Bleaching: 0.3 x amount of bleaching earth
  * Deodorization: 1.3 x (FFA + FAME)

- For CIE with 0.1% and post-bleaching with 0.5% BE
  * HIGH OIL LOSSES: 1.5% on incoming oil
Standard Chemical Interesterification
Enzymatic Interesterification

LIPOZYME TL IM

- Triacylglycerolhydrolase (also named Lipase)
- Obtained *Thermomyces lanuginosus* (*in past from pancreas*)
- Thermo-stable (max 75°C)
- Kosher and Halal approved

= Immobilized enzyme (on silica carrier)

- Stabilizes the enzyme
- Allows use in fixed bed reactors
- Gives higher enzyme life time (lower enzyme cost)
Enzymatic Interesterification Process

Feedstock: **Fully refined oil (blend)**

Enzyme is inactivated by oxidation products and acids. Enzyme is not sensitive for FFA and water.

### Required feedstock quality

- **Moisture**: max. 300 ppm
- **Soaps**: max. 5 ppm
- **Peroxide value**: max. 1 meq O₂/kg
- **p-anisidine value**: max. 5
- **Water extract of oil**: 6.0 < pH < 9.0
Enzymatic Interesterification Process

• Fixed bed reactors
  Oil flow: top-to-bottom or bottom-to-top

• Oil flow rate
  EIE is a slow reaction: approx. 1000 x slower than CIE
  1.0 – 1.7 kg interesterified oil/kg enzyme
  Feedstock dependent (conversion of high melting fats is slower)

• Enzyme productivity
  2.5 – 4.0 ton interesterified oil/kg enzyme
Enzymatic Interesterification Process

- **Post-bleaching**
  Not required for most of the EIE oil (98 %)
  Oil for enzyme conditioning (4 kg oil/kg enzyme) has high FFA content (± 5%) and high soap content (1000 ppm) and needs to be reprocessed
  First EIE oil after enzyme conditions (20 ton/500 kg enzyme) still has 200-300 ppm soaps. It is recommended to collect this soapy oil in a separate buffer tank and bleach it before blending it with the bulk of the EIE oil

- **Post-deodorization**
  EIE oil requires only mild deodorization (no off-flavors, minimal FFA formation)
Standard Enzymatic Interesterification
Enzymatic Interestesterification: Industrial plant

EIE plant – Skid mounted

4 x 500 kg

(48-80 ton/d)
Chemical versus Enzymatic Interesterification

Palm Stearin / Soybean Oil (70/30 w/w)

SFC (%) vs Temperature (°C)

- Initial Blend
- Deo CIE Blend
- Deo EIE Blend

Biggest Delta SFC
Delta SFC ~ 10%

IUPAC 2.150 non tempered
Chemical *versus* Enzymatic Interesterification

![Graph showing the relationship between temperature and solid fat content for different materials.](attachment:image.jpg)

**Solid Fat Content [%]**

**Temperature [°C]**

*Palm stearin/Coconut oil (60/40)*

IUPAC 2.150 non tempered
Chemical versus Enzymatic Interesterification

**Chemical interesterification**

- Gives fully random TAG composition (determined by FA composition)
- Highly reproducible and cost-effective
- Easy (batch)
- Suitable for frequent feedstock changes

**Chemical interesterification**

- Hazardous catalyst → safe handling required
- Side reactions require proper post-treatment
- High oil losses due to side-reactions (soap and FAME formation)
- Risk of flavour reversion & reduced stability
- Loss in minor-components (e.g. antioxidants)
Chemical versus Enzymatic Interesterification

+ Enzymatic interesterification

- Simple, clean and safe process.
- No side reactions giving higher interesterified oil yield;
- Better quality (less DAG, better oxidative stability) than CIE
- Lower capital investment cost compared to CIE

- Enzymatic interesterification

- Feedstock quality is critical for good enzyme activity
- Enzyme activity is feedstock dependent
- Operating cost largely determined by enzyme cost (enzyme activity)
Oil Modification Processes

COST COMPARISON

Fractionation

Enzymatic IE
2.5 ton oil/kg enzyme

Chemical IE
0.1% CH₃ONa

Hydrogenation
△ IV 50

Cost in Europe (EUR / ton)
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Hydrogenation – Still a future?

Only full hydrogenated fats (IV < 1) are potential feedstock for zero *trans* fats.

But, full hydrogenated fats mostly have a too high melting point and can therefore not be used *as such*.

Full hydrogenated fats can be used in oil blends for interesterification, but then this will have a high cost.

Real potential of full hydrogenation will mainly depend on feedstock cost (e.g. Sunflower oil vs palm oil).
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CONCLUSIONS

Dry fractionation is a powerful process to produce many different zero trans fats starting from a given feedstock (mainly palm and palm kernel oil).

Continuous fractionation opens the door for further more economical processing on large scale.

Enzymatic and chemical interesterification allow to further develop zero trans fats with the same functionality as trans-rich partial hydrogenated fats.

Still a lot of product development work can be done by combining full hydrogenation, dry fractionation and interesterification to arrive at better zero trans fats of the future!!
Thank you for your attention!!!

Science behind technology!

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