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# RANDOM ENZYMATIC INTERESTERIFICATION

INDUSTRIAL PLANT

Enzyme reactors

PILOT PLANT

LAB REACTOR

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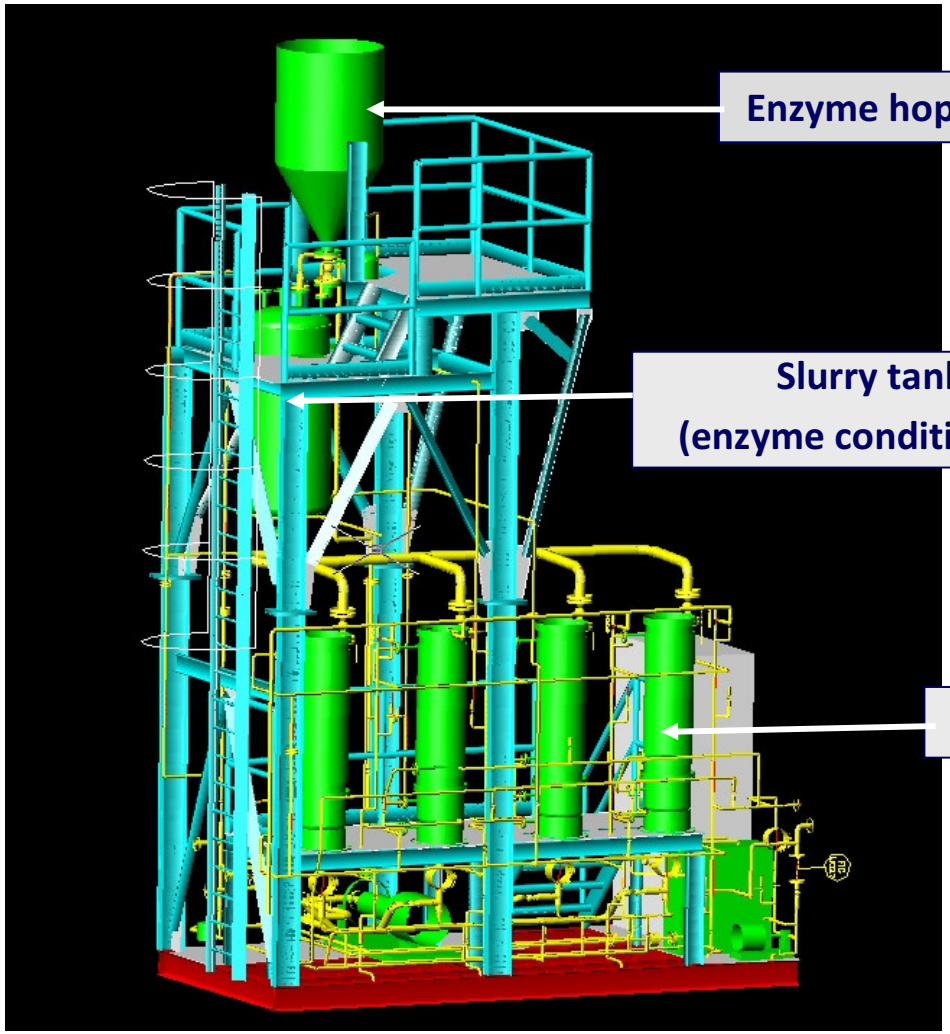
# RANDOM ENZYMATIC INTERESTERIFICATION



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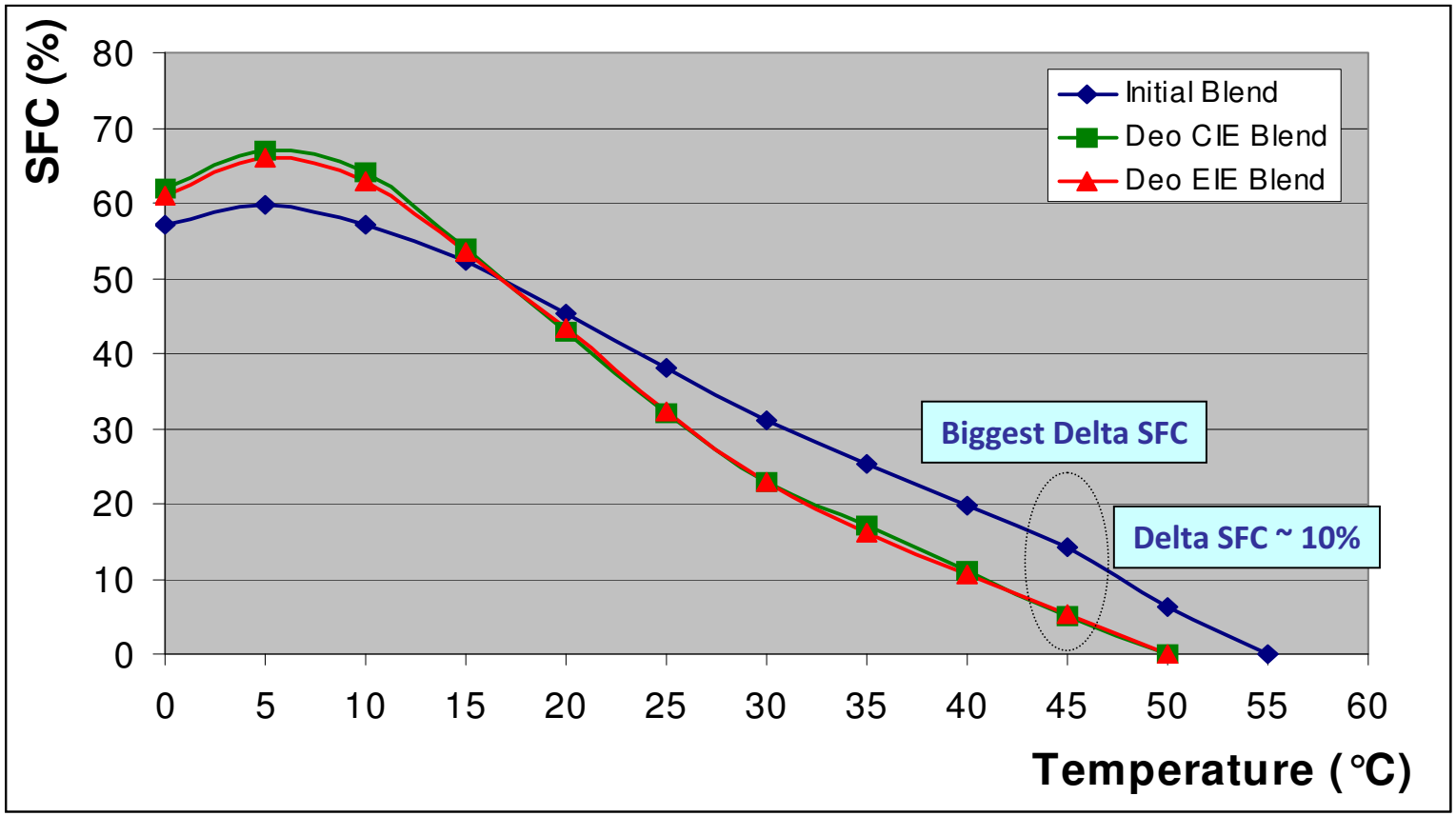
# RANDOM ENZYMATIC INTERESTERIFICATION

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




IUPAC 2.150 non tempered

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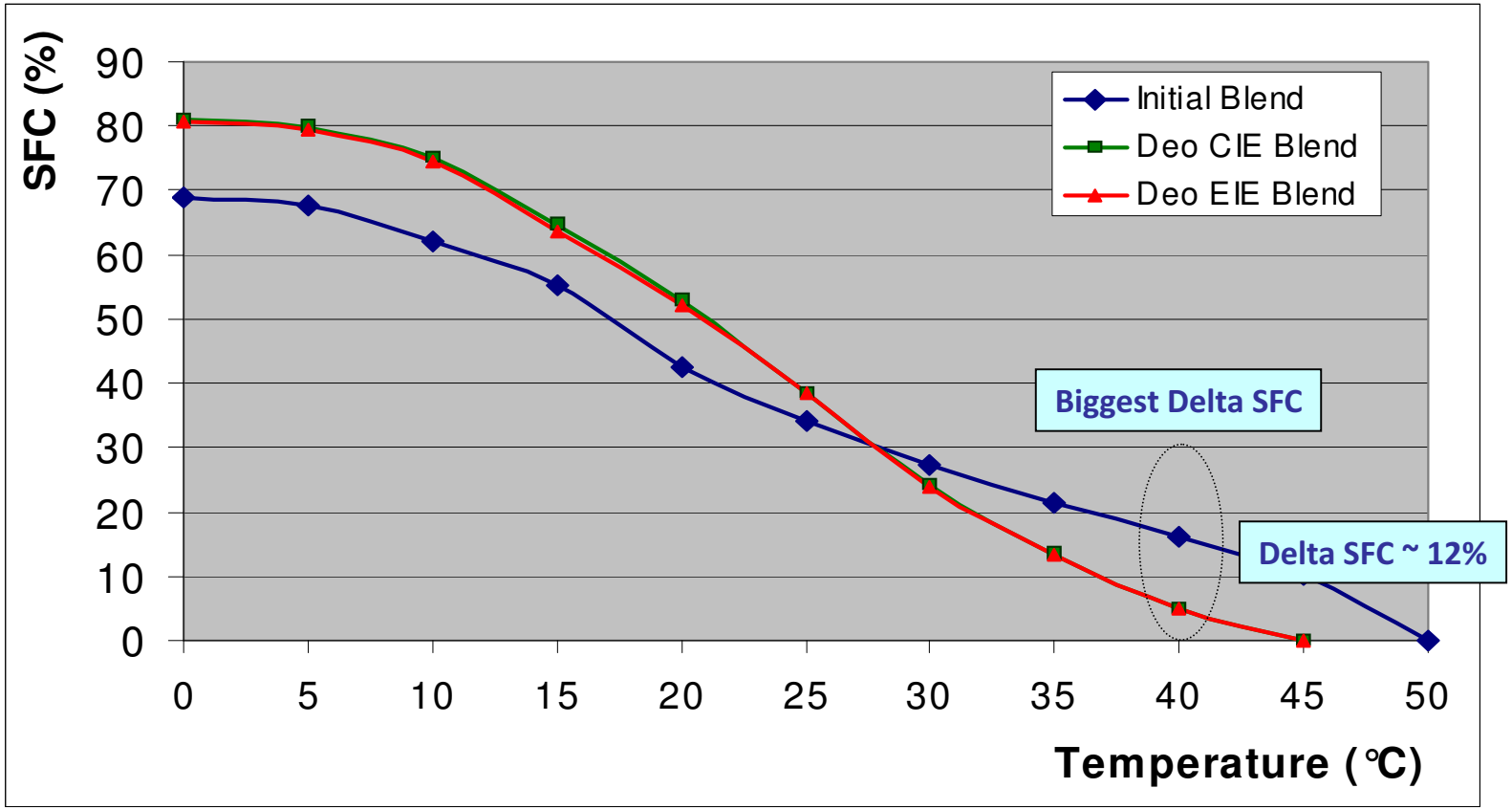
# RANDOM ENZYMATIC INTERESTERIFICATION

## Palm Oil / Fully Hydrogenated Palm Oil/ Palm Kernel Oil (40/30/30 w/w/w)




IUPAC 2.150 non tempered

# RANDOM ENZYMATIC INTERESTERIFICATION

## Calculation of the degree of interesterification (D.I.) during a continuous process

- Measure the SFC profile (by p-NMR) of the initial blend and of the EIE and/or CIE blend (produced on lab-scale).
- Select the temperature where the SFC difference between initial and IE blend is the highest (in the example before: 40°C).
- Calculate the Δ SFC: Δ SFC full conversion (in the example before: 10%).
- Regular sampling at the outlet of each enzymatic reactor (once a day).
- Calculate the Δ SFC (at 40°C): Δ SFC current conversion.

Calculate the degree of interesterification (D.I) as follow:

D.I. = 100*( Δ SFC current conversion/ Δ SFC full conversion)

D.I. is used to adjust the flow-rate

D.I. is used to verify that the enzyme is still active and able to assure full conversion in the used operating conditions

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# RANDOM ENZYMATIC INTERESTERIFICATION

## Enzyme conditioning (slurry tank)

- **De-aeration step (air removal):**

**Immobilized enzyme is suspended and surface-wetted with the oil at 70°C, slow stirring, oil/enzyme ratio typically 3/1.**

**Vacuum is created in the tank and enzyme suspension is agitated continuously during a period of ~30 min.**

- **Conditioning step (water removal):**

**The suspension is transferred by gravity from the slurry tank to the EIE reactor.**

**Recirculation starts from the EIE reactor back to the slurry tank (~60 min.).**

**When conditioning time is elapsed, oil is pumped back to storage till low level in the slurry tank and the EIE reactor is ready for EIE.**

**At early stages, increase of EIE oil acidity (FFA) and soaps that progressively disappear -> FFA of EIE oil ~ 0.2% / soaps in EIE oil: N.D.**

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# RANDOM ENZYMATIC INTERESTERIFICATION

## Compositional properties of EIE blend

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

| TAG composition (% by HPLC) | Initial Blend | EIE Blend |
|---|---|---|
| StStSt | 21.5 | 12.6 (---) |
| StUSt | 31.0 | 38.7(++) |
| StUU | 26.6 | 36.9 (+++) |
| UUU | 20.9 | 11.9 (---) |
| LLL [A] | 7.0 | 1.6 |
| PPL [B] | 6.0 | 14.1 |
| SFC % at highest Δ | 14.9 | 5.1 |

St: saturated fatty acid on glycerol

U: unsaturated fatty acid on lycerol

P: Pamitic Acid ; L: Linoleic Acid

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# RANDOM ENZYMATIC INTERESTERIFICATION

## Quality parameters of EIE blend (Palm Stearin / Soybean Oil (70/30 w/w))

| | Initial Blend | Deo EIE Blend | Deo CIE Blend |
|---|---|---|---|
| FFA (% C16:0) | 0.03 | 0.02 | 0.02 |
| **Tocos (ppm)** | **462** | **321** ◄···► | **175** |
| α-tocopherol | 77 | 60 | 30 |
| α-tocotrienol | 37 | 30 | 16 |
| γ-tocopherol | 212 | 151 | 85 |
| γ-tocotrienol | 65 | 47 | 31 |
| δ-tocopherol | 61 | 30 | 13 |
| δ-tocotrienol | 10 | 3 | - |
| **DAG (%)** | **3.6** | **3.6** ◄···► | **4.9** |
| Colour (R/Y 5 ¼) | 2.4/24 | 2.2/23 | 1.7/20 |

- **The tocopherol/tocotrienol content was better retained (higher oxidative stability) in the EIE blend.**
- **The DAG content was less in the EIE blend (less oil degradation).**
- **The EIE blend doesn't need to be post-bleached: no colour increase/fixation like in CIE.**

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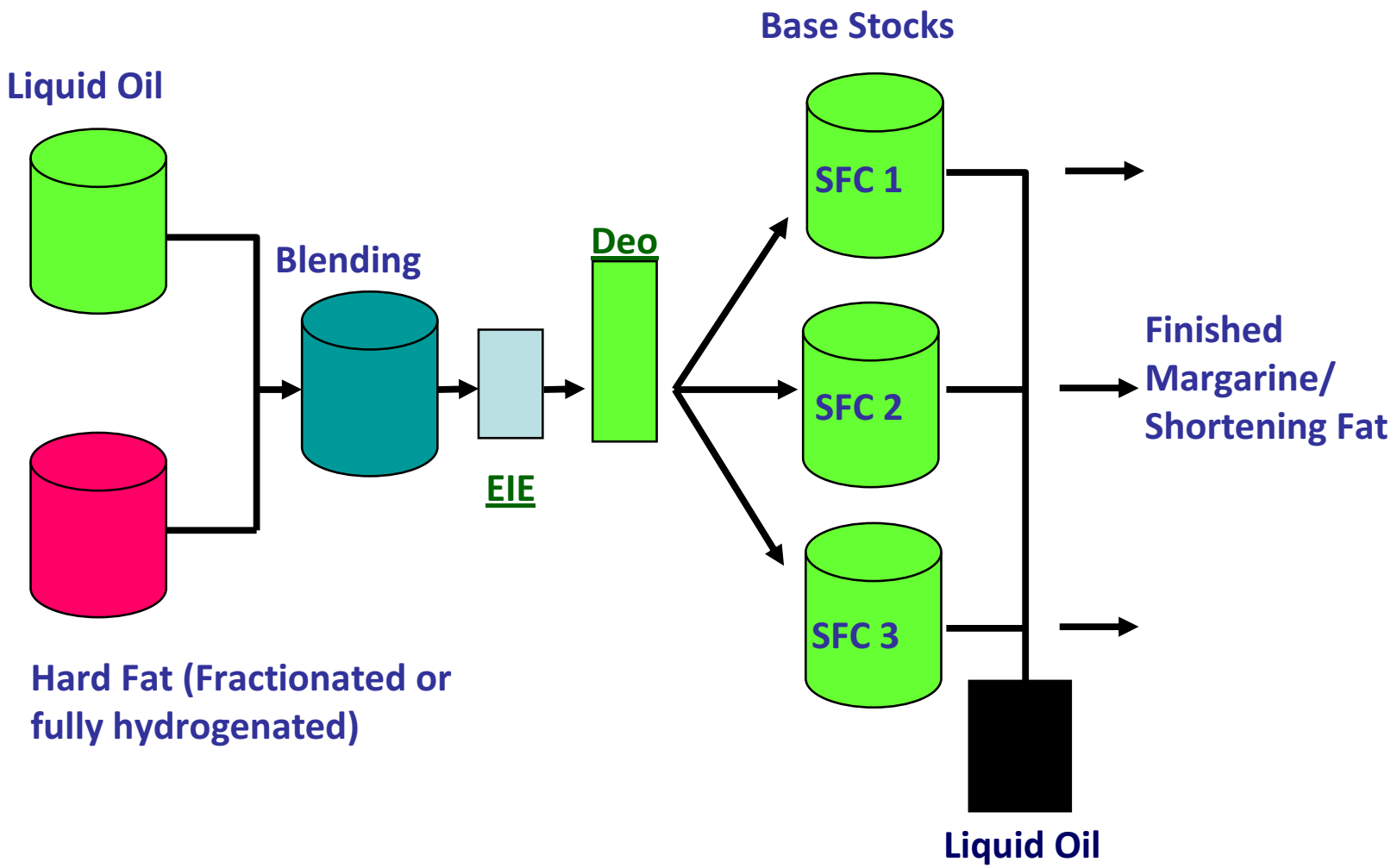
# RANDOM ENZYMATIC INTERESTERIFICATION



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# COCOA BUTTER EQUIVALENTS

## Confectionery fats - High StUSt fats for CBE : blends of tropical fats and fractions

| TAG composition (% by HPLC) | SOS (%) | POS (%) | POP (%) |
|---|---|---|---|
| Cocoa Butter | 26 | 37 | 18 |
| CBE 1 (HPMF/ Shea Butter Stearin) | 48 | 14 | 38 |
| CBE 2 (HPMF/ Shea Butter Stearin/ Illipe) | 43 | 24 | 33 |
| CBE 3 (HPMF/ Illipe) | 34 | 15 | 50 |
| CBE 4 (HPMF/ Sal Stearin/ Illipe) | 43 | 21 | 36 |

S: Stearic Acid; P: Pamitic Acid ; O: Oleic Acid

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# REGIO-SELECTIVE ENZYMATIC INTERESTERIFICATION

## High StUSt fats: CBE

## Example 1: High oleic oil/stearic acid (acydolysis) or stearic methyl ester (ester/ester exchange)

**RM IM**

| TAG composition (% by HPLC) | High oleic oil | After acidolysis (1/1) |
|---|---|---|
| SSS | N.D. | 5 |
| SOS | N.D. | 20 |
| POS | 0.5 | 5 |
| POP | 1 | 1 |
| SOO | 7 | 35 |
| POO | 12 | 2 |
| OOO | 68 | 15 |
| Others | ~ 12 | ~ 17 |
| StUSt (SOS+POS+POP) | 1.5 | 26 |
| TAG Oil/FA or FAME | - | 1/1 |

Residual FA (FAME)

S: Stearic Acid; P: Pamitic Acid ; O: Oleic Acid; St: saturated fatty acid; U: unsaturated fatty acid

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# REGIO-SELECTIVE ENZYMATIC INTERESTERIFICATION

**High StUSt fats: CBE**

## Example 2: Soft PMF/stearic acid (acydolysis) or stearic methyl ester (ester/ester exchange)

**RM IM**

| TAG composition (% by HPLC) | Soft PMF | | After acidolysis (1/1) |
|---|---|---|---|
| SSS | < 0.5 | | 4 |
| PSS | N.D. | | 6 |
| PPS | < 0.2 | | 5 |
| SOS | 1.5 | ····▸ | 14 |
| POS | 9 | ····▸ | 30 |
| POP | 47 | ····▸ | 15 |
| SOO | 2 | | 6 |
| Others | ~ 40 | | ~ 20 |
| StStSt (SSS+PSS+PPS) | < 1 | ····▸ | 15 |
| StUSt (SOS+POS+POP) | 58 | ····▸ | 59 |
| TAG Oil/FA or FAME | - | | 1/1 |

**Residual FA (FAME)**

S: Stearic Acid; P: Pamitic Acid ; O: Oleic Acid; St: saturated fatty acid; U: unsaturated fatty acid

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# REGIO-SELECTIVE ENZYMATIC INTERESTERIFICATION

## High StUSt fats: CBE

## Example 3: High oleic oil/high stearic oil (ester/ester exchange)

**RM IM**

| TAG composition (% by HPLC) | Blend 50/50 | After EIE |
|---|---|---|
| SOS | N.D. ⋯⋯→ | 25 |
| POS | 0.5 ⋯⋯→ | 10 |
| POP | 0.5 | 0.5 |
| StUSt (SOS+POS+POP) | 1 | 35.5 |

S: Stearic Acid; P: Pamitic Acid ; O: Oleic Acid; St: saturated fatty acid; U: unsaturated fatty acid

## Conclusions (ex. 1, 2 and 3)

- Enzymatic acidolysis ends with a quantity of FA that must be stripped.
- Enzymatic acidolysis of a high oleic oil (low StUSt) principally increases the SOS content.
- Enzymatic acidolysis of a soft PMF (high StUSt, high POP) re-adjust the SOS/POS/POP ratio.
- EIE of high oleic/high stearic oils (low StUSt) principally increases the SOS content.
- The StStSt content is significantly increased: further fractionation (multi-step) is mandatory to fit with CB compositional properties.

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# TAKE HOME MESSAGES

- Interesterification (CIE or EIE)

  Low *trans or trans* free margarines and shortening fats.
- EIE is preferred: « bio-process », no chemicals, low temperature, less oil losses, improved quality of the EIE oil.
- EIE with:

  - a non selective (random) lipase (TL IM) for « Margarines and Shortening Fats ».
  - a sn 1,3 regio-selective lipase (RM IM) for « Functional Lipids » [CBE …].
  - industrial plant configuration is identical (fixed bed).
- EIE is a continuous process:

  - preferably running on bulk fats.
  - quality of the feed oil is important to keep the process competitive.
  - no post-bleaching.
- The D.I. is typically followed based on the SFC profile (p-NMR) of the EIE oil.
- Production of CBE: blends of oleic and stearic feeds and regio-selective enzyme: increase of SOS and POS; increase of StStSt (post-fractionation to improve compositional properties). Intermediate stripping of FA (FAME).

**Enzymatic Processing and Modification - Current and Future Trends**

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Het Pand, University of Ghent, Belgium

# Science behind Technology

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