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Biochemistry, nutrition and technology of lipids. Partie II

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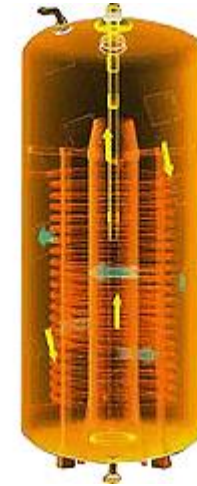
HAL Id: hal-02786344

<https://hal.inrae.fr/hal-02786344v1>

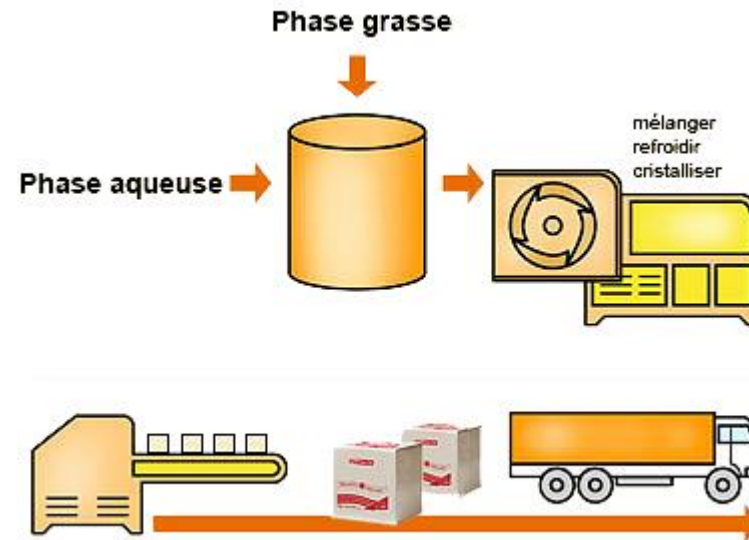
Submitted on 4 Jun 2020

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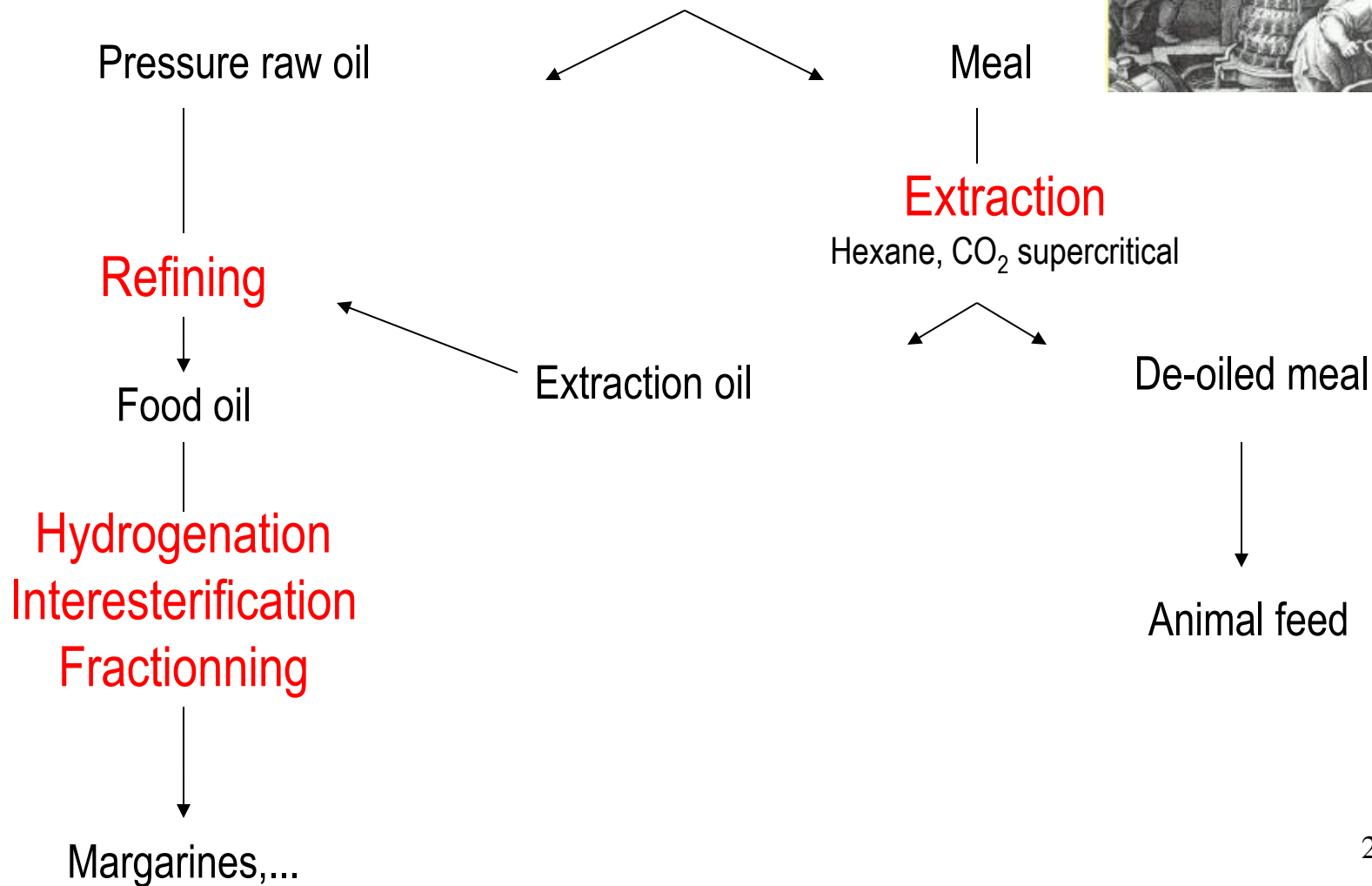


4. TECHNOLOGY OF VEGETABLE OILS

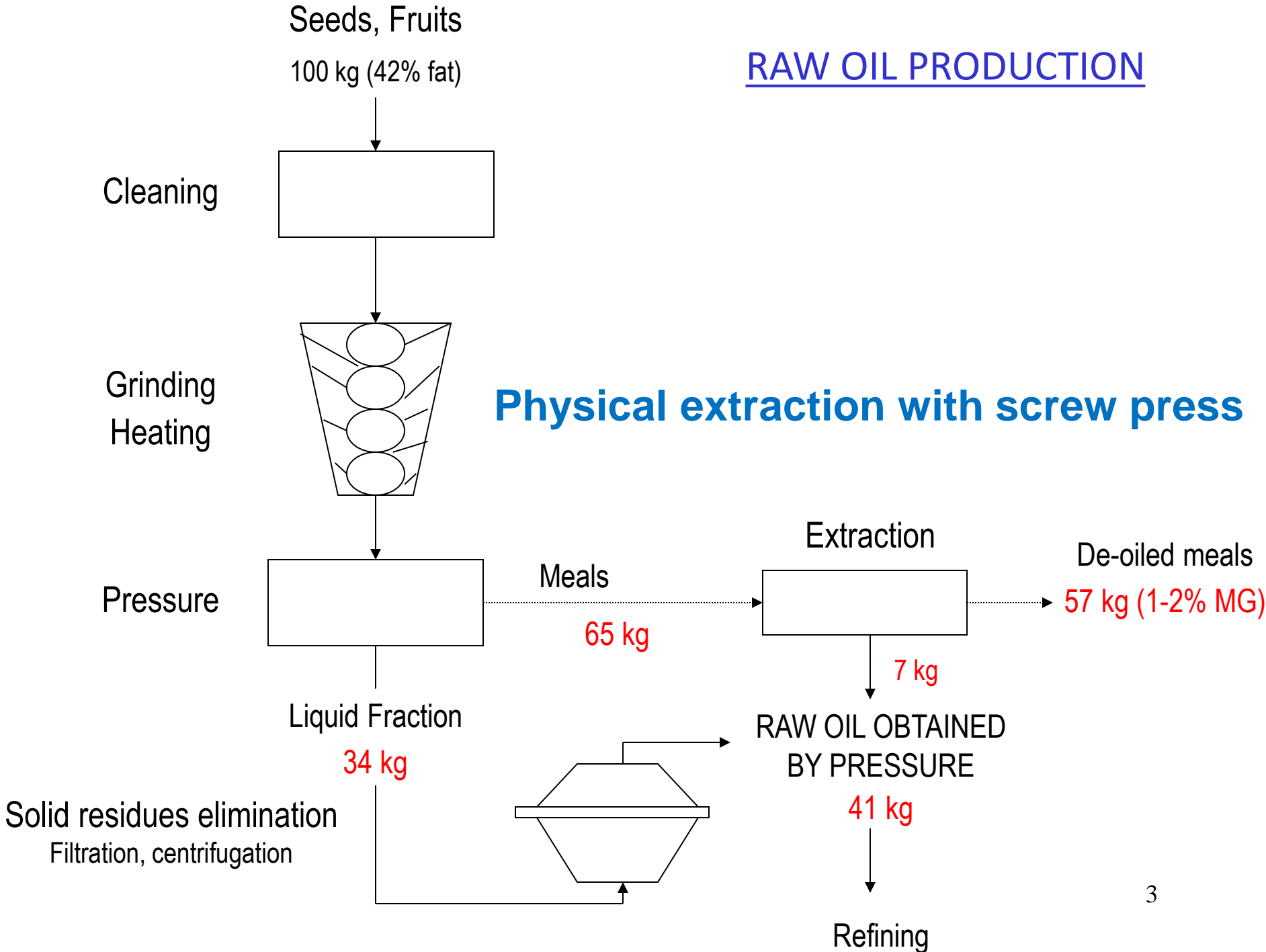


TECHNOLOGY OF VEGETABLE LIPIDS

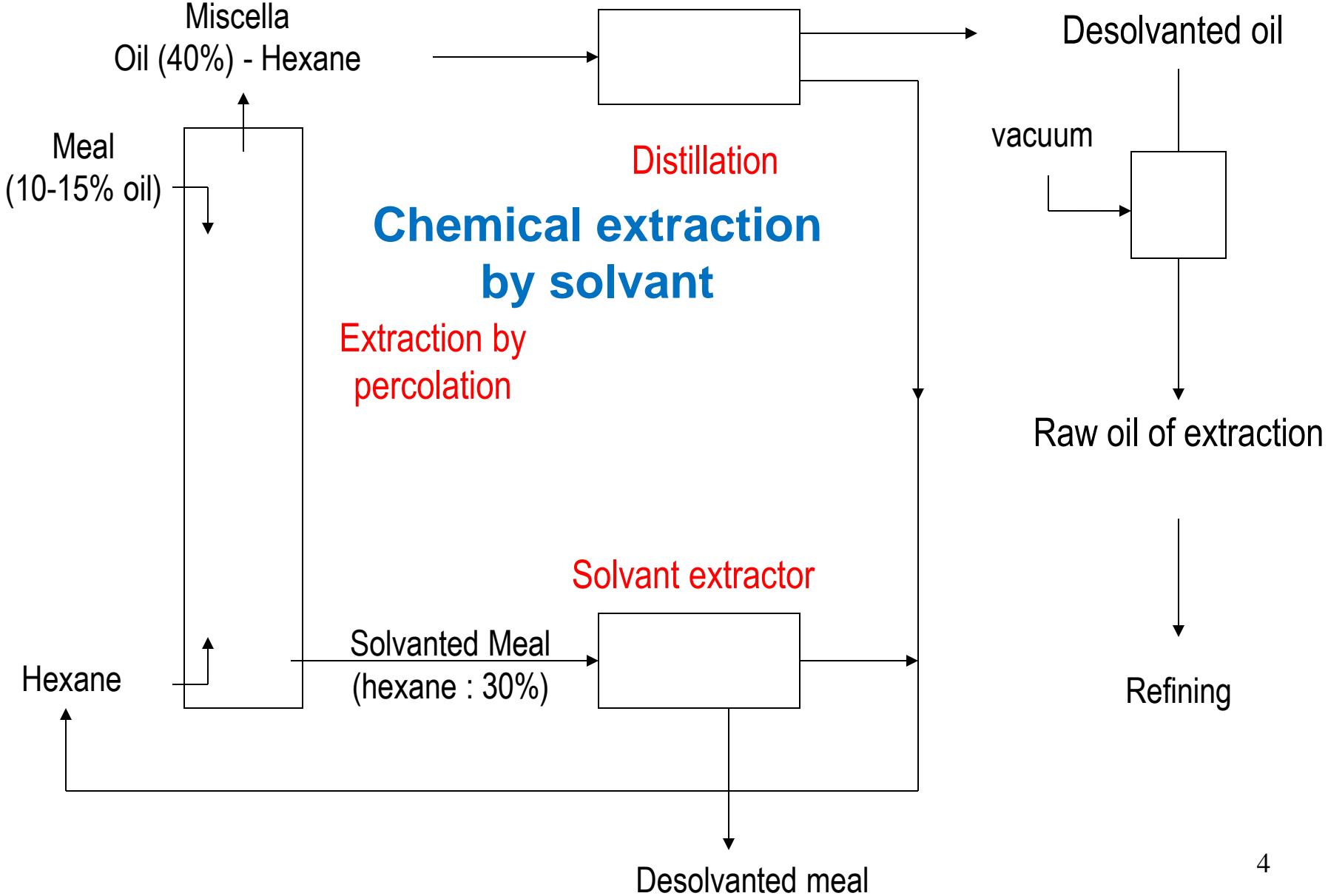
Seeds, Grains, Fruits
Control, Cleaning, Dehulling
Grinding, heating, pressing, separation



RAW OIL PRODUCTION



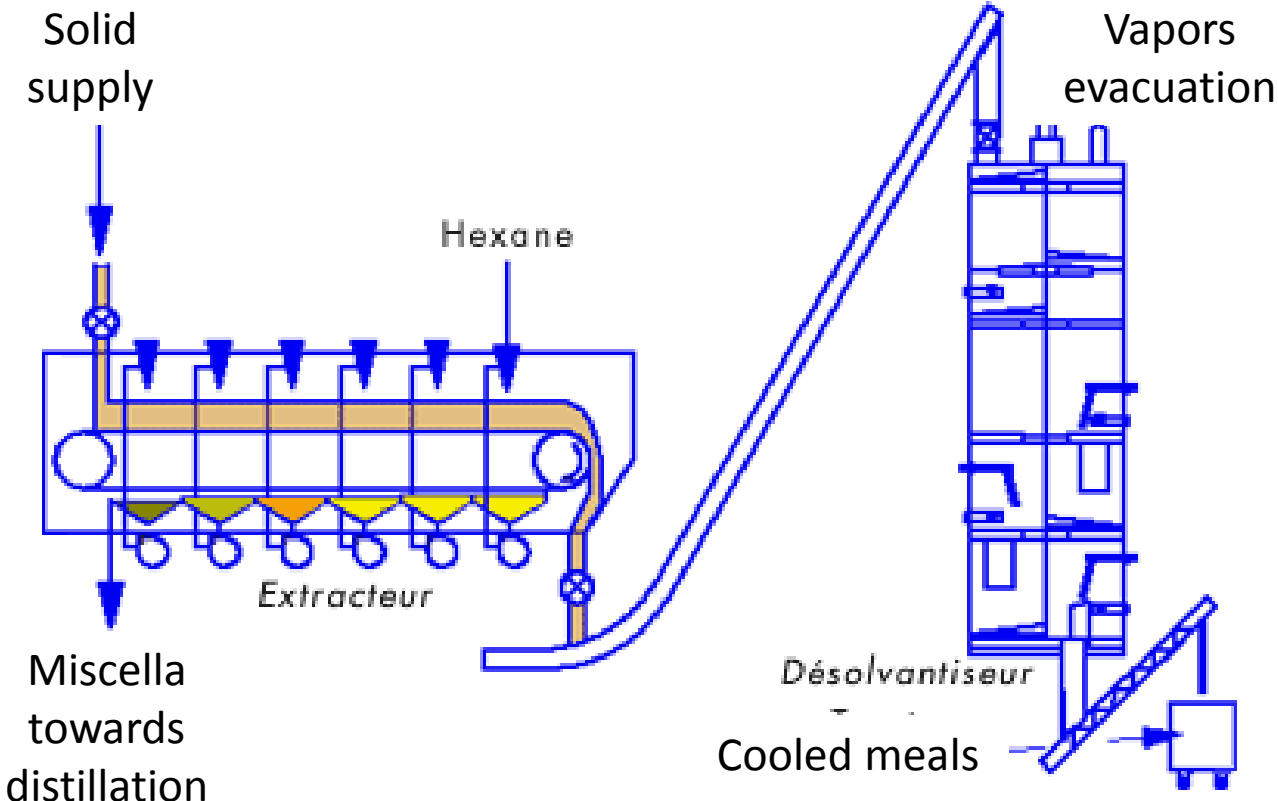
EXTRACTION



Hexane extraction plant



SAIPOL Le Mériot Pant (photo C. Helsly)



REFINING OF FOOD FATS



Unwanted Constituents



NATURE	TENEUR	ORIGINE	EFFET
AG libres	0,5 – 5%	Hydrolyse (naturel)	Goût Fumée
P.lipides	0,2 – 1,5%	Naturel	Trouble instabilité Organoleptique
Produits d'ox.	f(Mat lère)	Auto-ox.	Instabilité Organoleptique Nutrition
Flaveurs	< 0,1%	Naturel Auto-ox.	Odeur Goût
Cires	≈ 100 mg/kg	Naturel	Trouble
Pigments	≈ 10 mg/kg	Naturel	Goût Couleur
Métaux	≈ 1 mg/kg	Naturel	
Contamin.	≈ 10 mg/t		santé

REFINING OF FOOD FATS



Rapseed
oil

Raw oil
Meal

Refined oil

Diester

Raw
Glycerine

REFINING

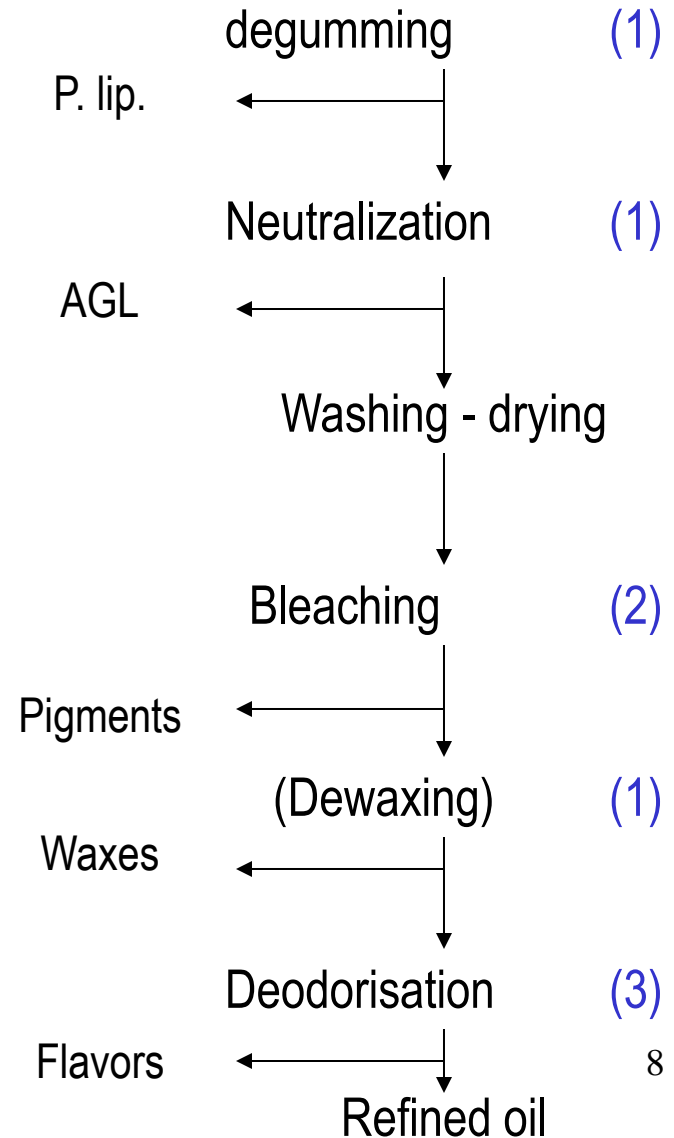
Objectives

- Get rid of undesirable const.
- Respect food grade application
→ strict reglementation

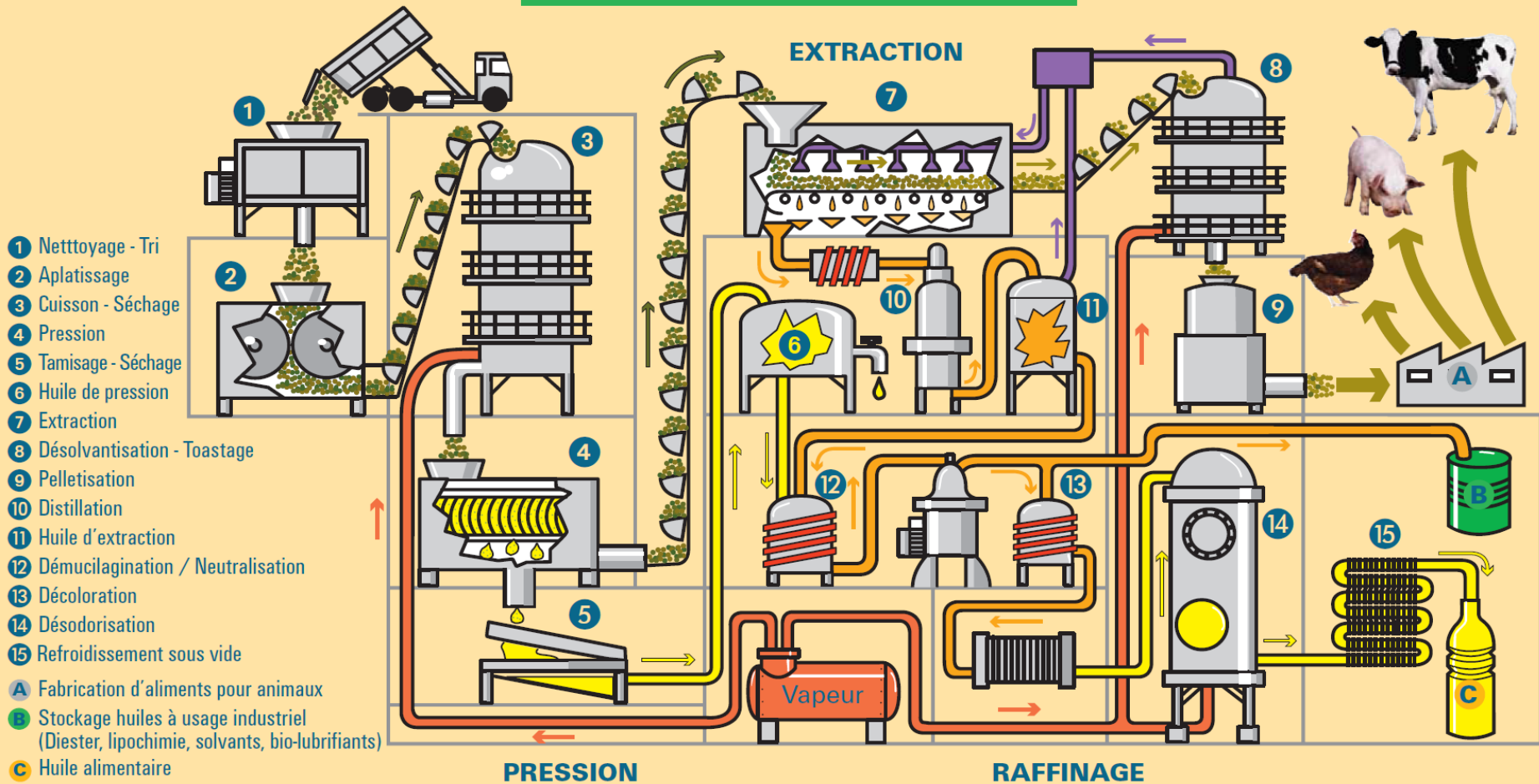
Principes:

- insolubilisation (1)
- adsorption (2)
- distillation (3)

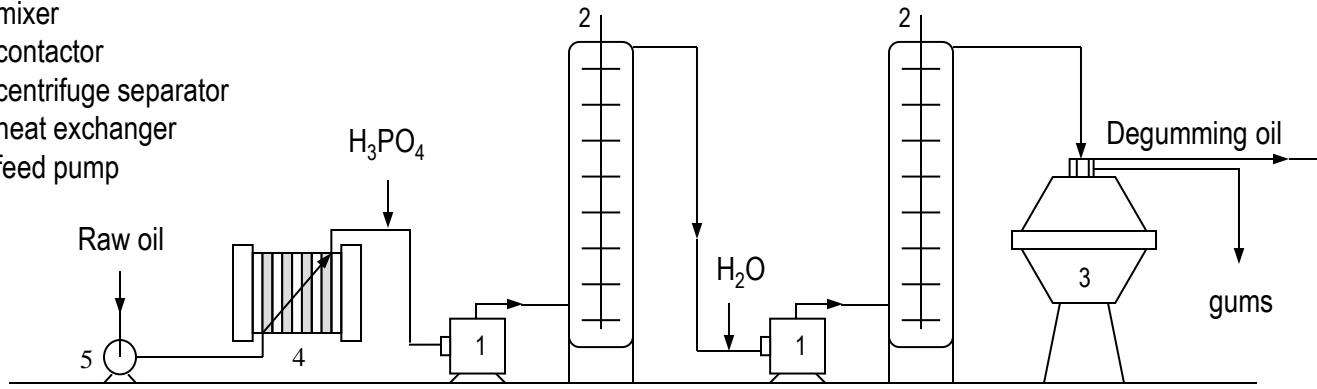
Technology



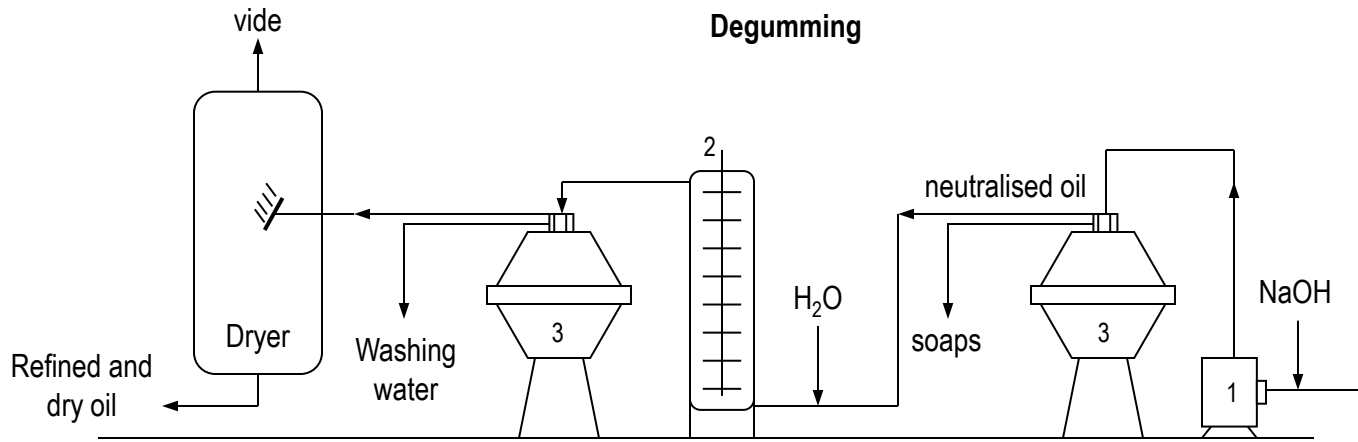
Transformation des graines oléagineuses



- 1- mixer
- 2- contactor
- 3- centrifuge separator
- 4- heat exchanger
- 5- feed pump



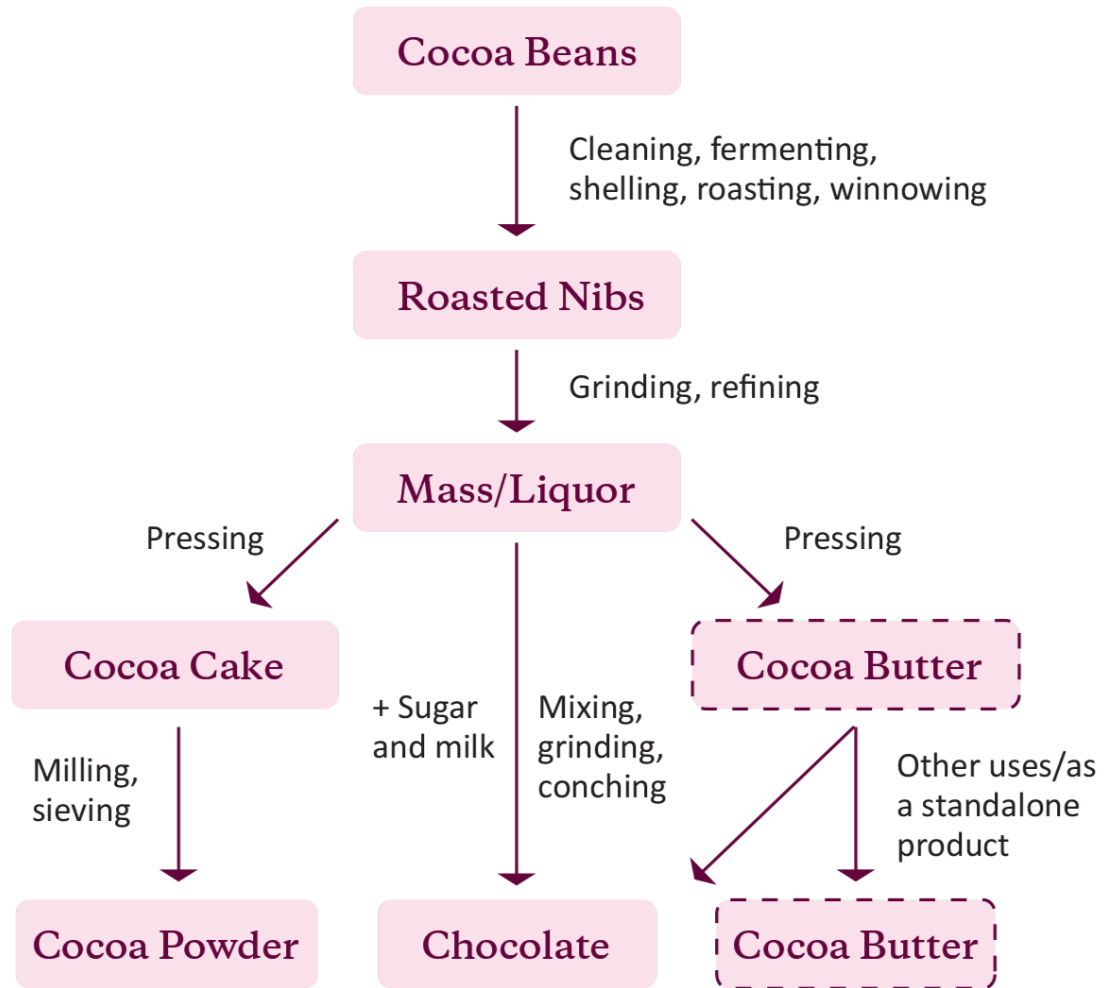
Degumming

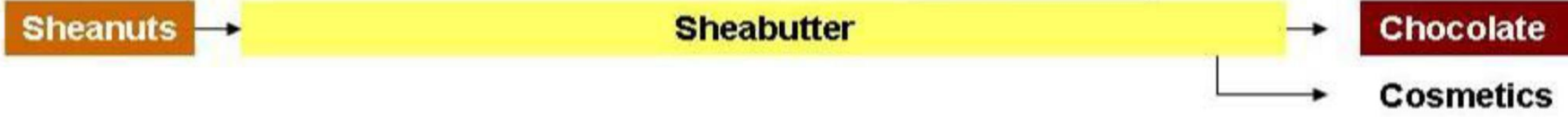
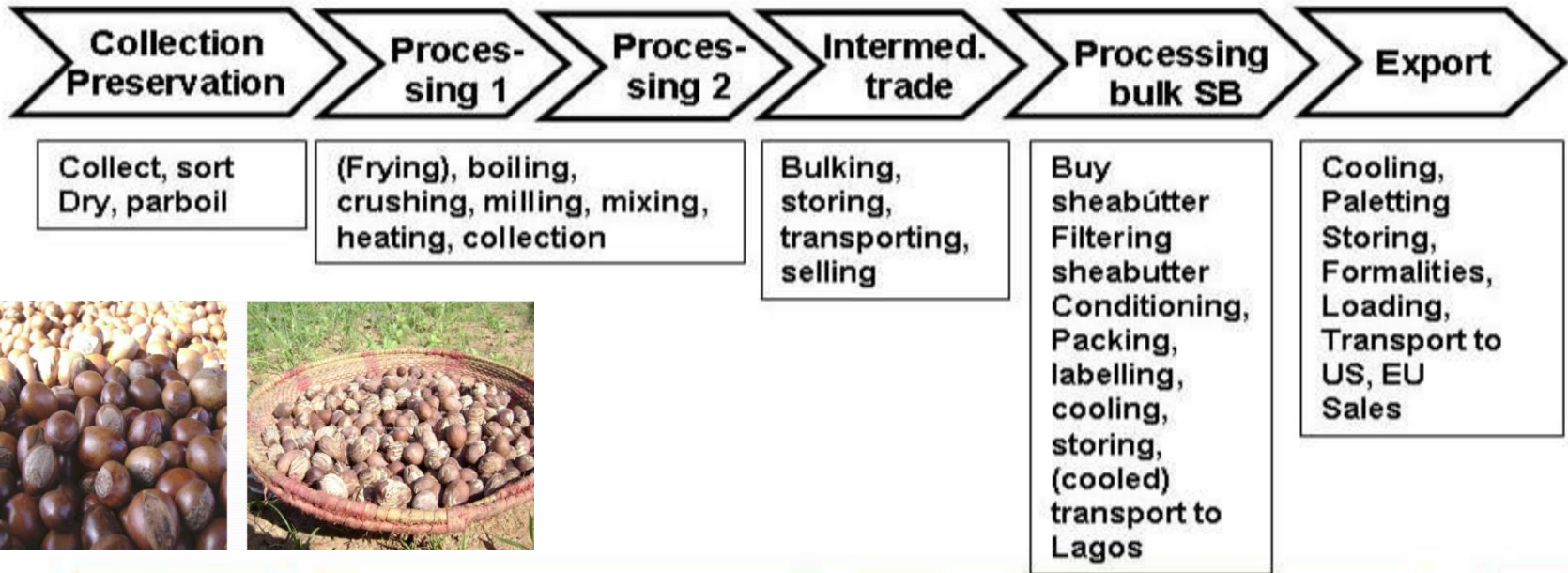


Drying

Washing

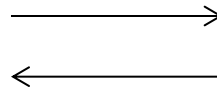
Decidification







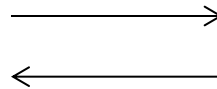
?



degumming

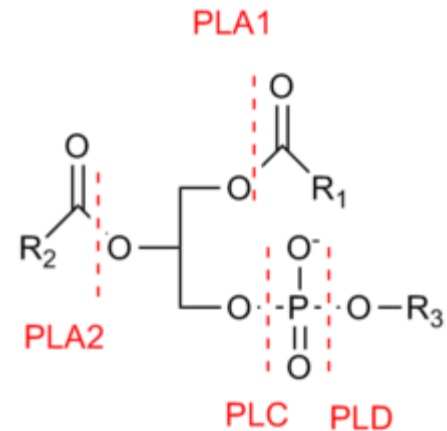


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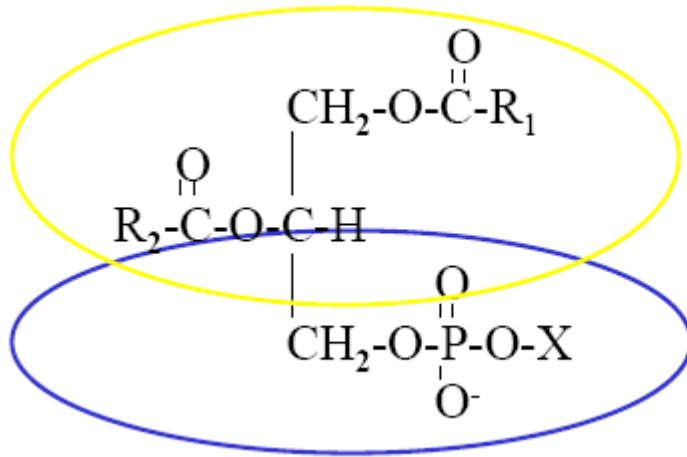
degumming

phospholipases: phospholipase A2 hydrolyses for instance fatty acid in position sn-2, releasing R2 FFA and a lysophospholipid. This lysophospholipid is a powerful detergent able to break cellular membranes. Snake and bee venoms are very rich in PLA2.

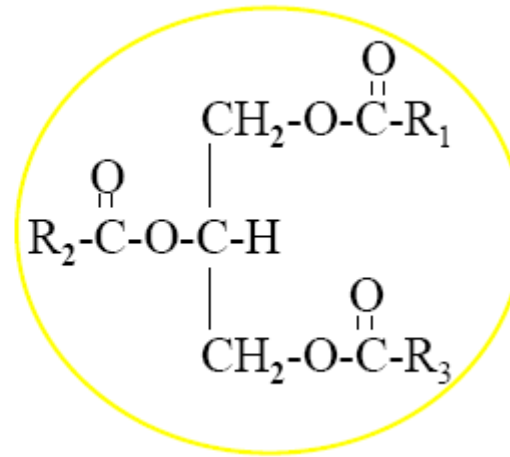


ENZYMATIC DEGUMMING

Phospholipids

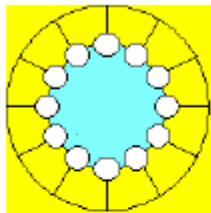


Triglycerides

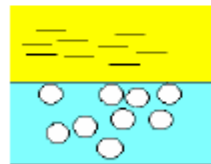


Affinity
for
water

Affinity
for
oil



● water ● oil
○ phospholipid



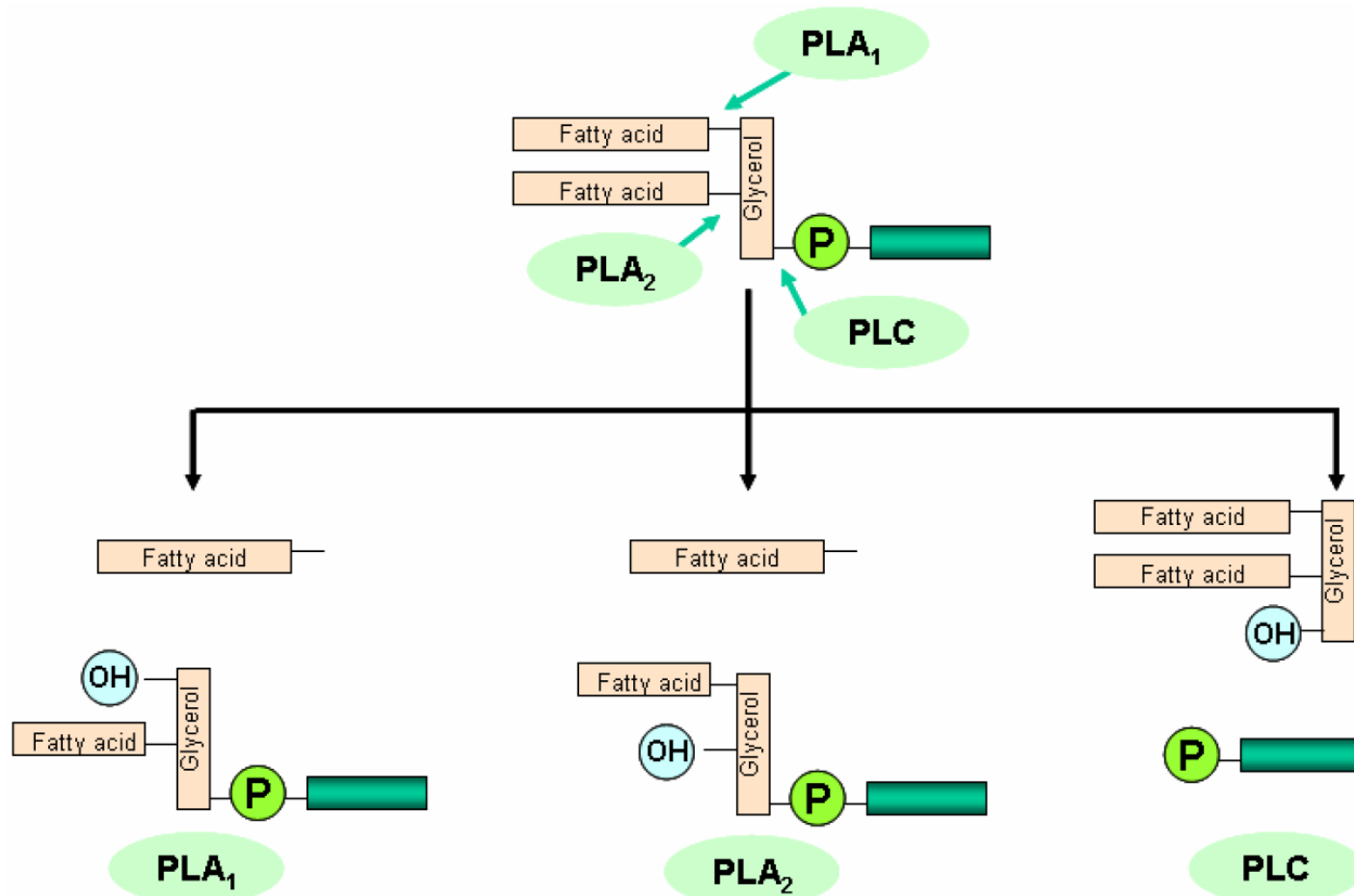
● water ● oil
○ phosphorous group



- In the conventional degumming and chemical refining process, gums work as emulsifiers and are responsible for the major part of the oil losses.

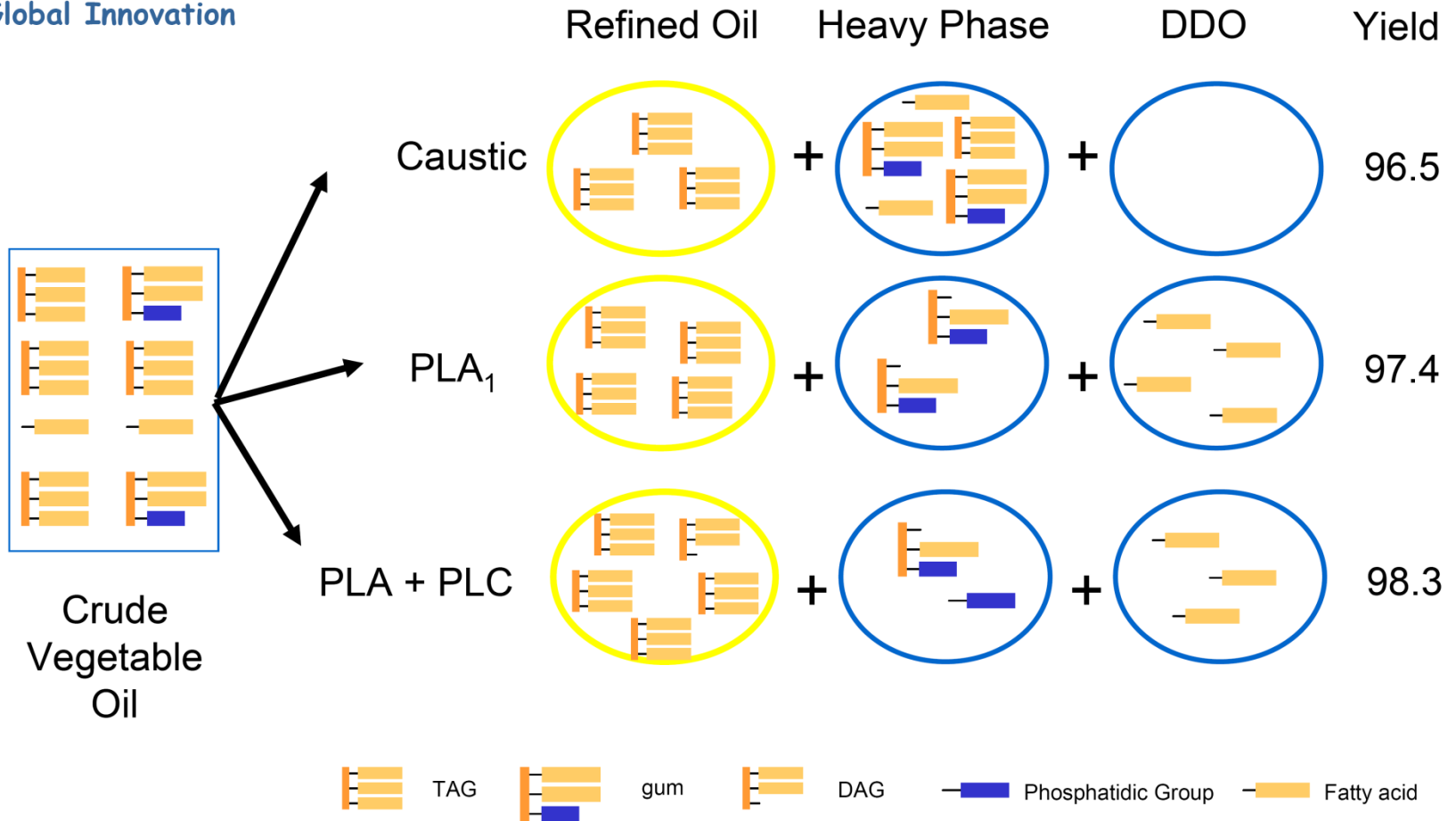
- In enzymatic degumming, the enzyme action eliminates the emulsification properties of the gums. The oil savings are proportional to the phosphorus (gums) in a ratio of 1 (oil) to 2 (gums).

ENZYMATIC DEGUMMING



ENZYMATIC DEGUMMING

Global Innovation



ENZYMATIC DEGUMMING

Commercial enzymes

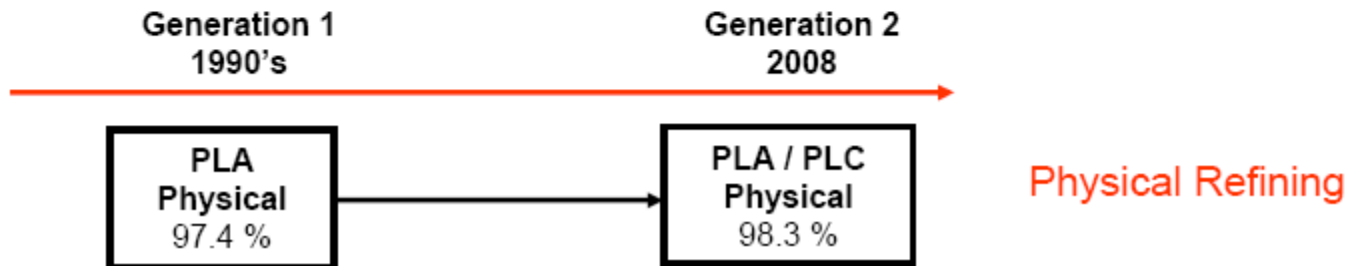
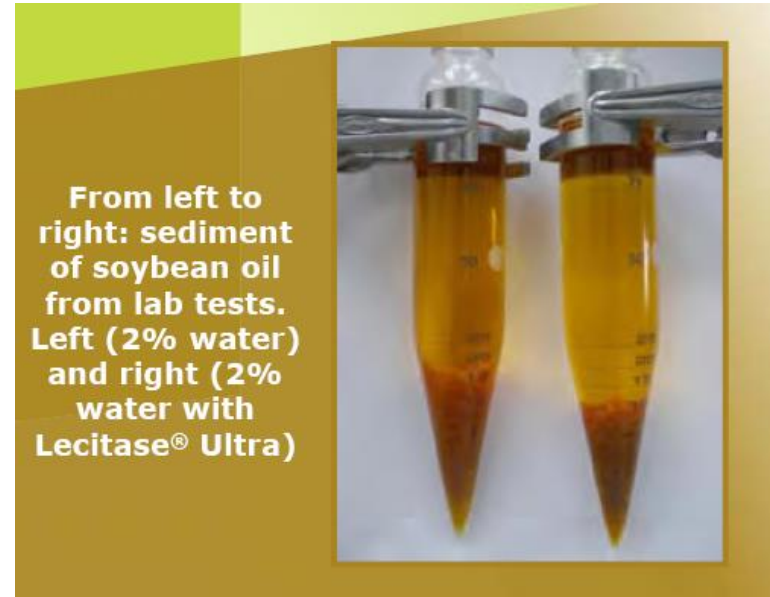
AB Enzymes' PLA₂, Rohalase[®] MPL

Dansico's PLA₂, FoodPro[™] LysoMax

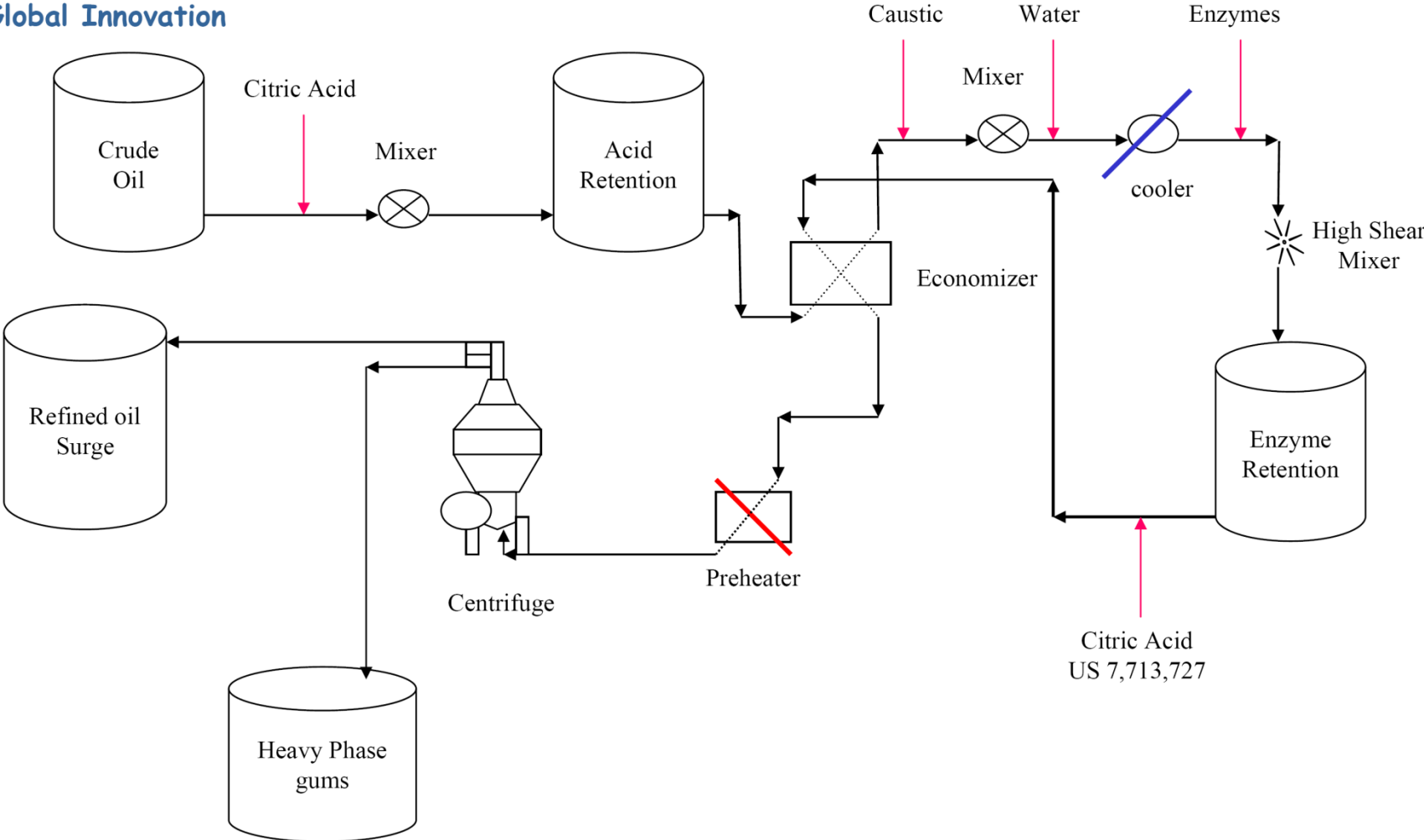
Novozymes' PLA₁, Lecitase[®] Ultra

Verenium's PLC, Purifine[™]

DSM's PLA₂, GumZyme[™]



Global Innovation

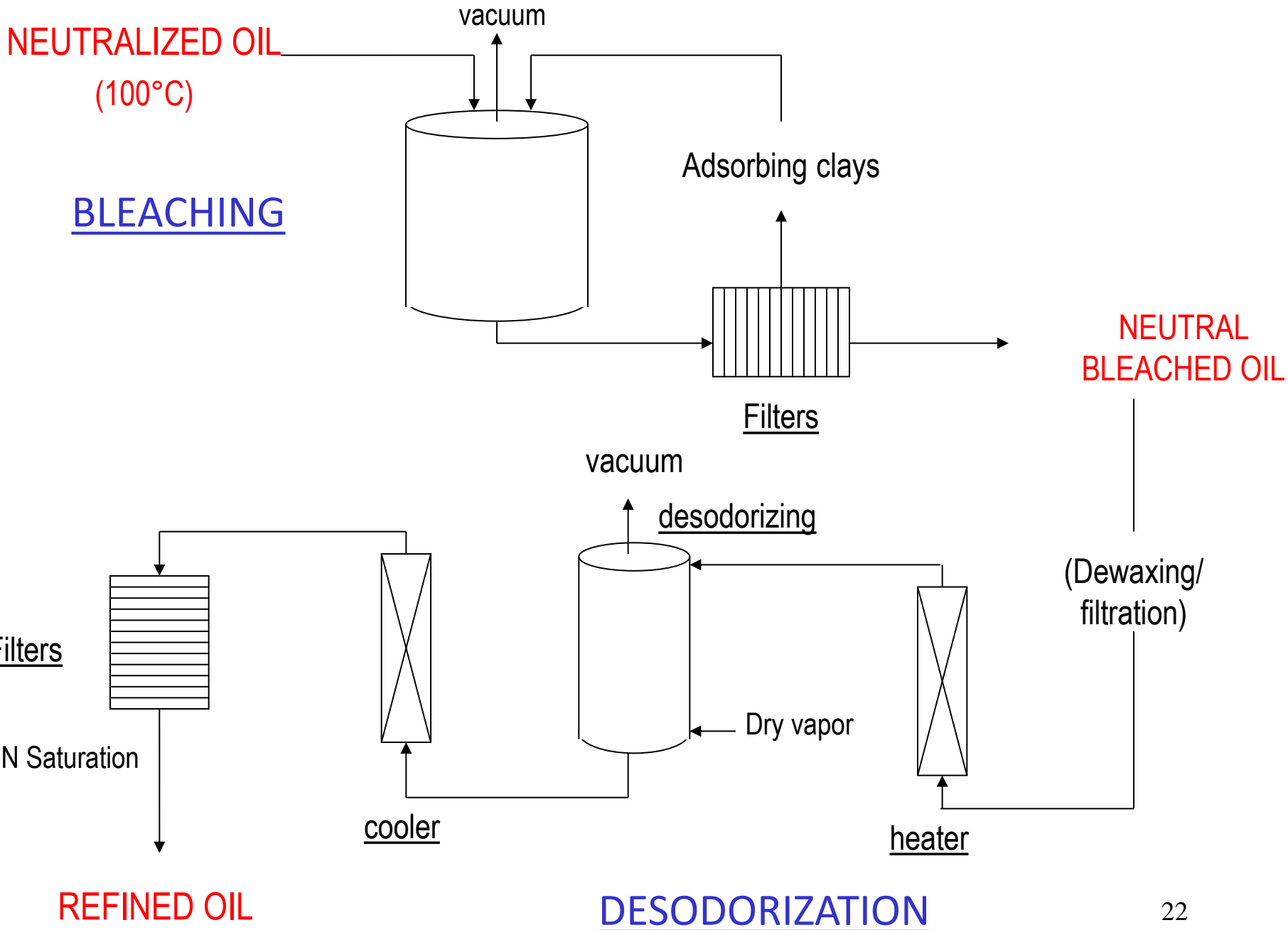


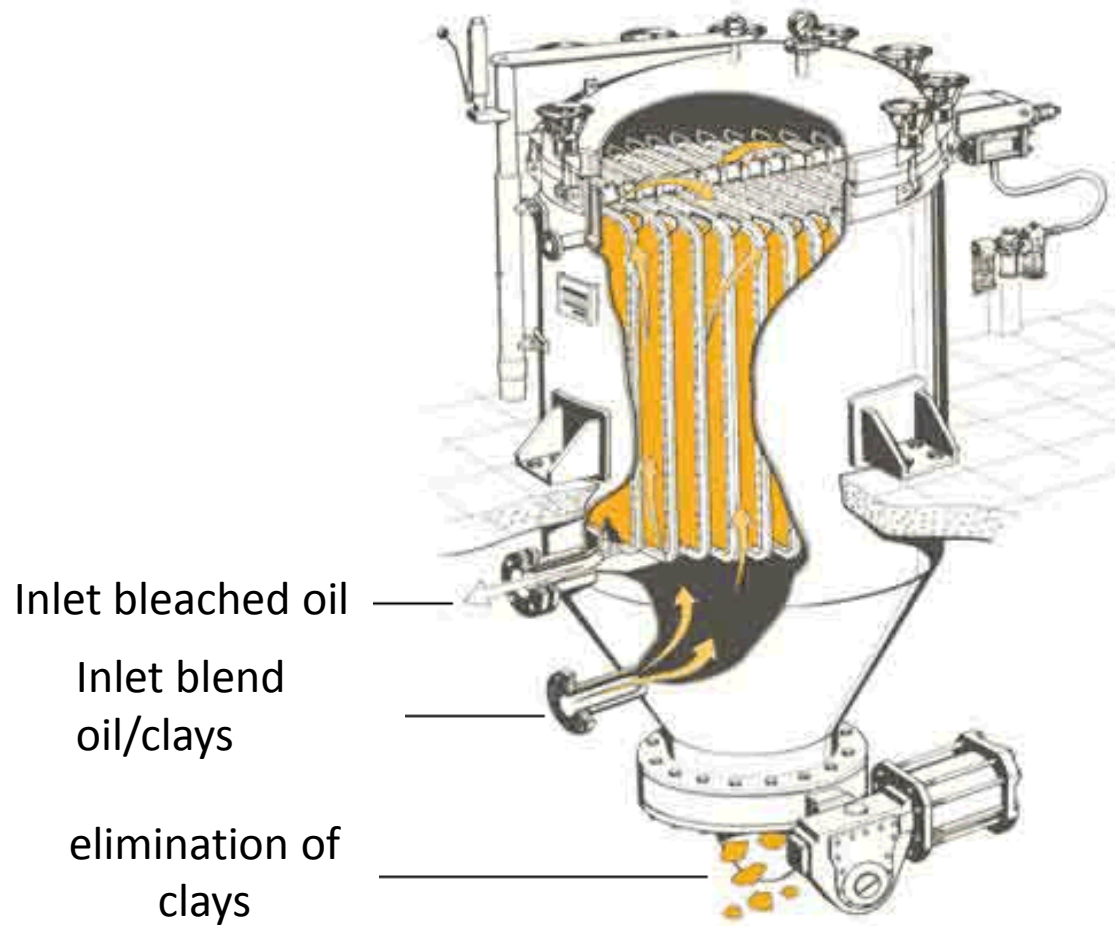
Neutralization

- Get rid of FFA under the form of soap or «**neutralization pastes**» : crucial unitary operation of refining. Pastes: soaps of sodium, soda, water, salts, sodium phosphates, mucilages, neutral oil can be retained, coloring constituents, oxydation products and diverse contaminants.
- Reactions taking place during this process are:
- ✓ neutralization of exces of phosphoric acid added to degum oils (1)
 - ✓ neutralization of FFA under the form of sodic soaps:
$$\text{R-COOH} + \text{NaOH} \rightarrow \text{R-COONa} + \text{H}_2\text{O} \quad (2)$$
- As weak acids, reaction in exces of alkali displaces equilibrium towards soaps formation, ↓ residual acidity. Excess ~ **0,01 et 0,05 %** for degummed soya oil
- AVOID «**side saponification**» (3), *i.e.* partial saponification of triglycerides => glycerol and sodium soaps. ↑ **losses.**
- Kinetics (1) & (2) >> (3)

Washing and drying

- **Get rid of alkalines substances** (soaps and soda in excess) present at the outlet of neutralization turbine, + last traces of metals, phospholipids and other impurities. Objectives: < 30 ppm of soap.
- **Good separation of raw oil/degumming** otherwise → important emulsions are being formed, difficult elimination of soaps.
- Washing more effective **if done in two steps, water washing must be as hot as possible (90°C).**
- **Post-stream Drying: humidity removal** if still present in washed oil before decoloring operation → risk of filter clogging specially in the presence of soaps.

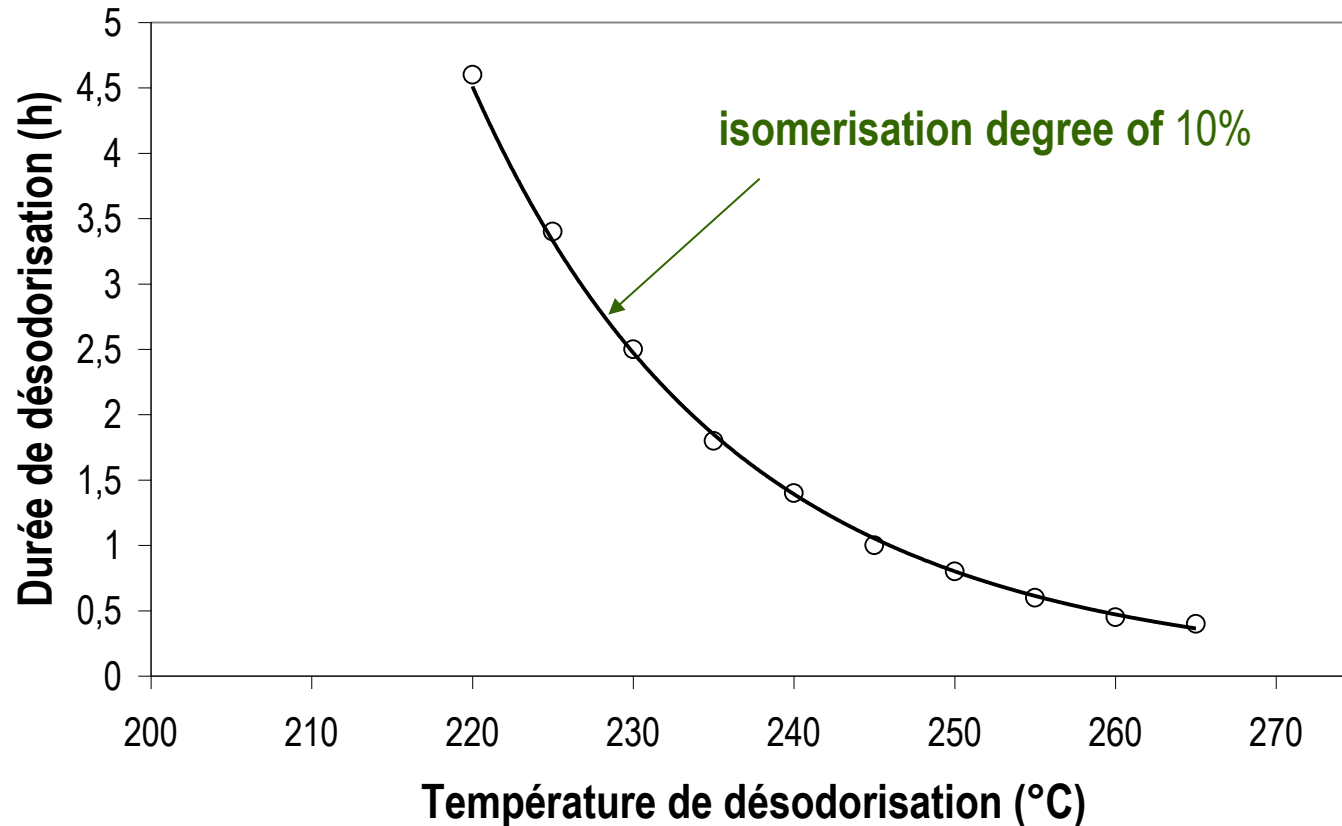




Vertical Filter

DESODORISATION

Effect of desodorisation parameters (couple temperature/length) on isomerisation degree of unsaturated acids (ex : linolenic acid C18:3)



Content in *trans* FA in refined oils (colza oils, combined oils) - Source ITERG

Teneur en AGT	Moyenne	Minimum	Maximum
En % des AG Totaux	0.8	0.3	1.9

REFINED FATS

Results

OIL	RAW	REFINED
TAG	85 – 89%	98 – 99%
P. Lipids	0,1 – 1,5%	< 50 mg/kg
FFA	0,5 – 5%	< 0,1%
Trans FA	< 0.1%	0.3 – 1.9%
Sterols	0 – 2% (soja)	< 1%
tocopherol	0,15%	0,1%
Pr. Ox	Variable	Variable
Waxes	≈ 100 mg/kg	< 5 mg/kg
Pigments	≈ 10 mg/kg	< 0,02 mg/kg
Metals	≈ 1 mg/kg	≈ 1 mg/t
Contaminants	≈ 10 mg/t	≈ 1 mg/t

ADAPTATION OF FATS to FOOD APPLICATIONS

- Objectives :*
- stability
 - technological, organoleptic and nutritional properties

Physical Levers :

Kinetics control of cooling (size and nature of fat crystals)

Fractionation (increase in FA with high or low melting points)

Chemical Levers

Hydrogenation (melting profile modif. [nature of FA] + ↗ stability)

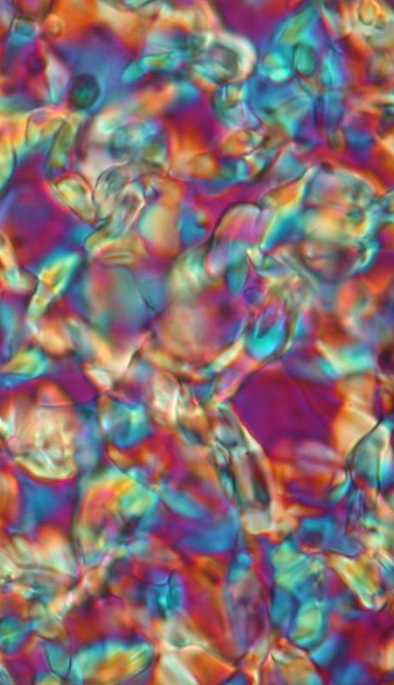
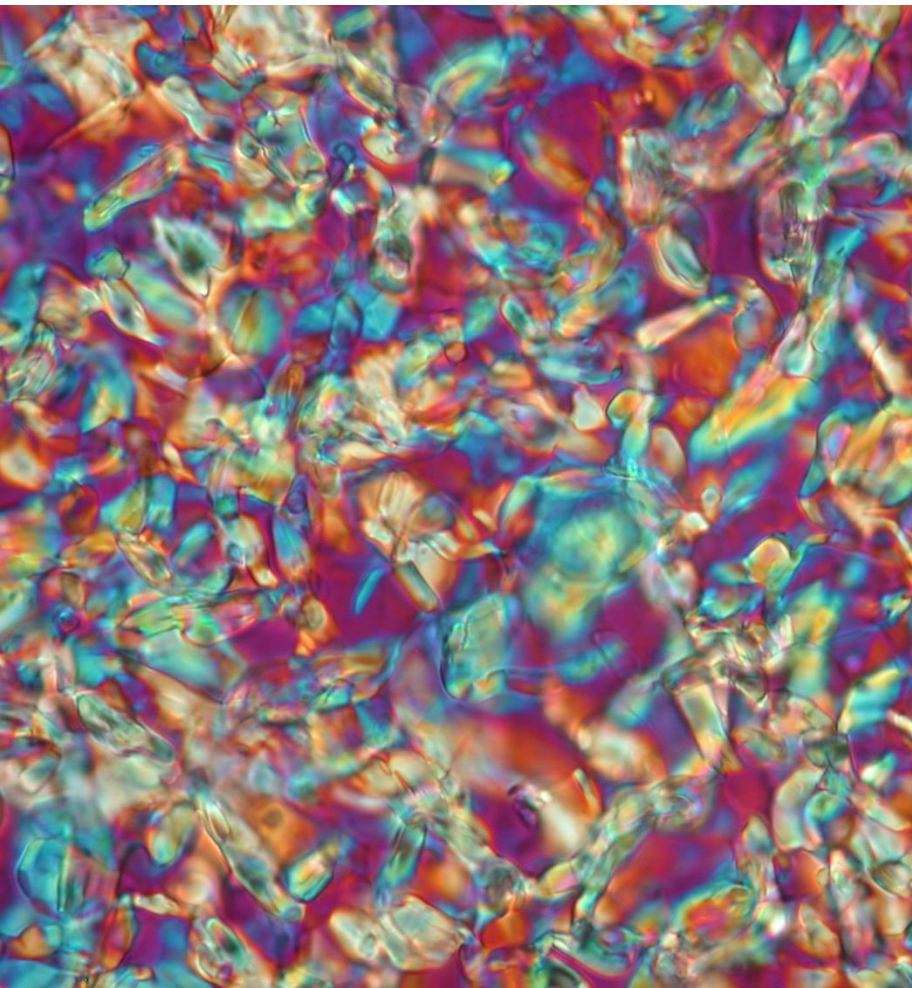
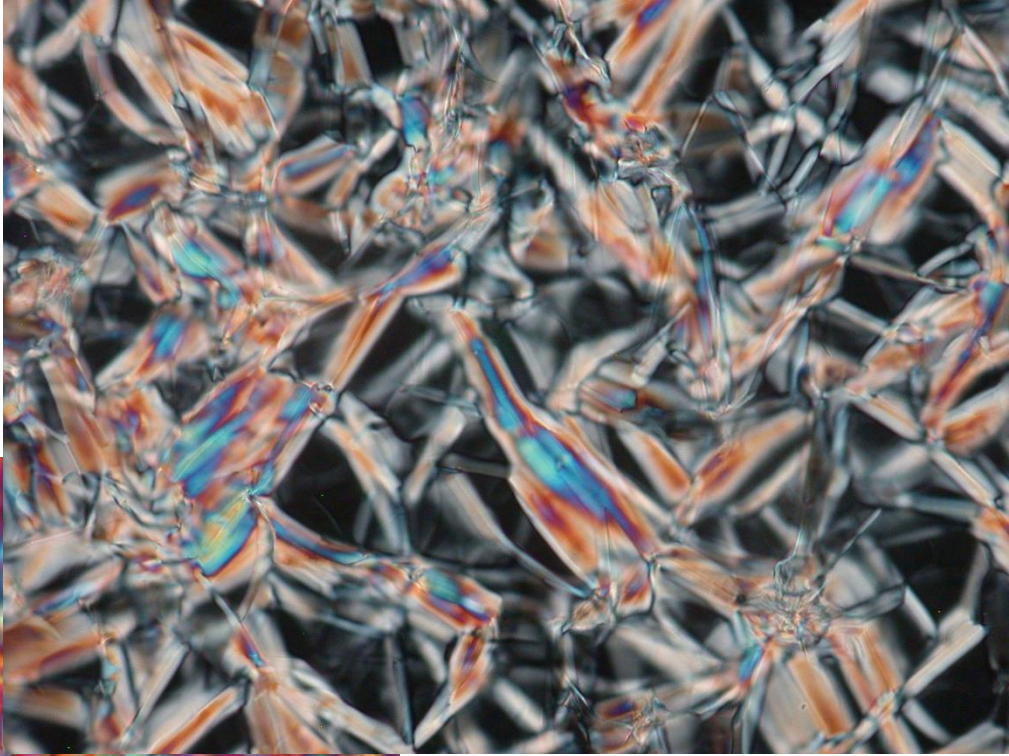
Interesterification (modif. Of melting profile [position of FA])

Enzymatic levers

Interesterification => structuration of TAG

glycerolysis => emulsifying power

Authorized treatments (arrêté du 12/02/73)



FRACTIONATION

FAT

← Composition

Melting

Equipments :

Parameters :

Cooling system →

Controlled cooling

← Cooling speed
overcooling.

Agitation speed

Cristallisation time

decanting
centrifugation
filtration →

separation

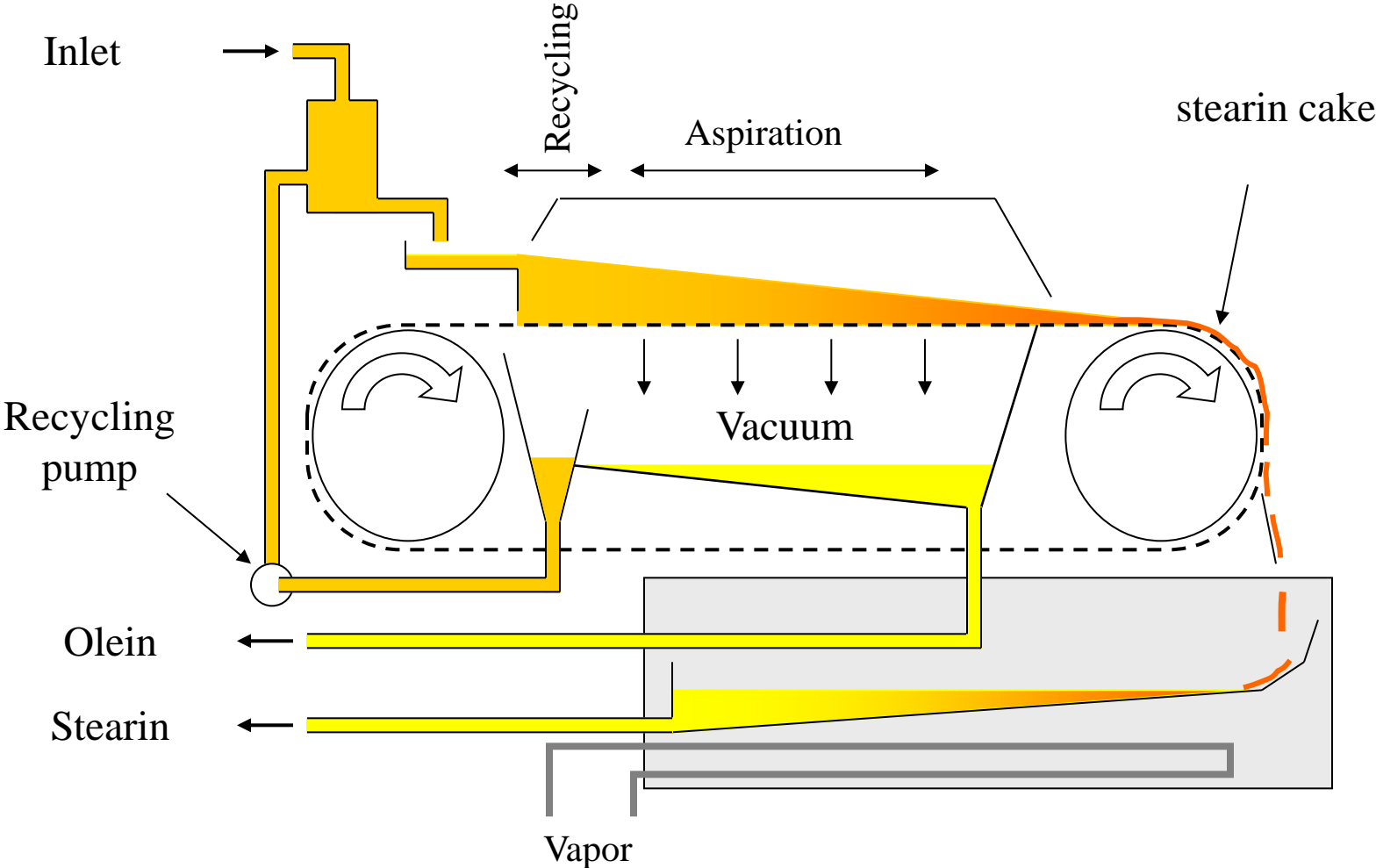
← temperature

HIGH melting point Fraction

LOW melting point Fraction

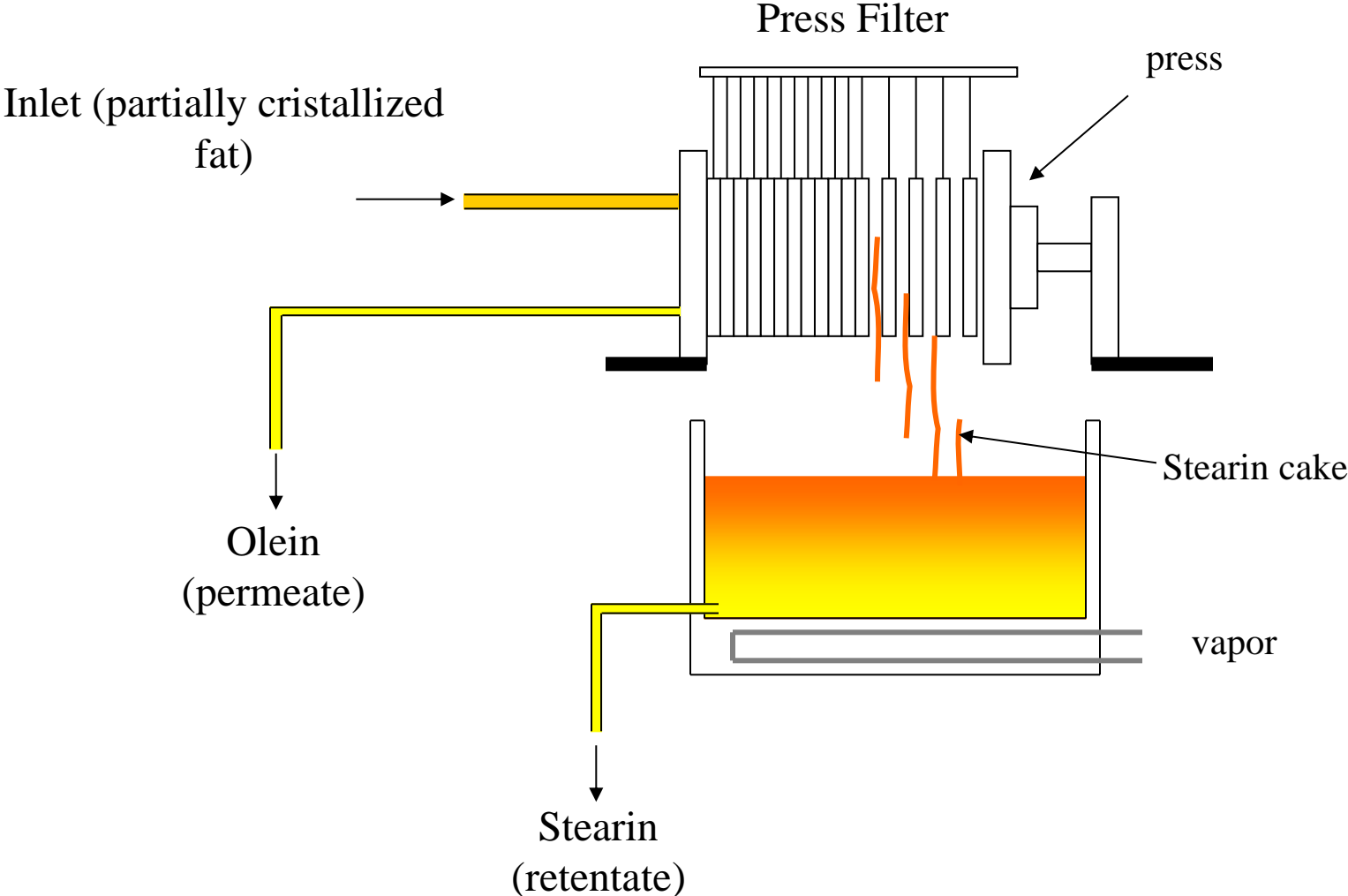
FRACTIONATION OF FATS

- Continuous Filtration -



FRACTIONATION OF FATS

- Press Filter -



FRACTIONATION OF PALM OIL

PALM

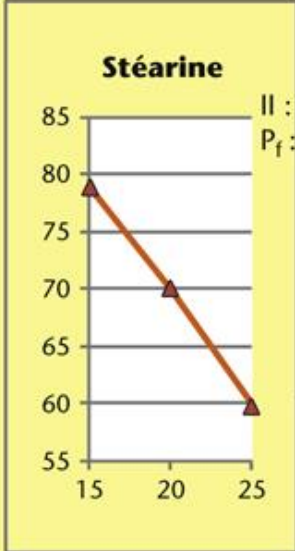
Composition moyenne	
Trisaturés (PPP)	6%
Disaturés monoinsaturés (PPO, POP)	50%
Monosaturés diinsaturés (POO, OPO)	40%
Triinsaturés (OOO)	4%

↓
Cristallisation (28°)

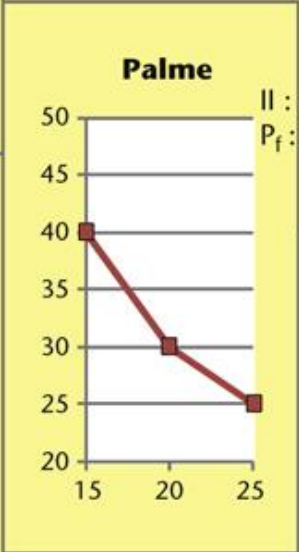
↓
Filtration

	Stéarine (20%)	Oléine (80%)
PPP	30%	0%
PPO, POP	26%	51%
OOP, OPO	44%	44%
OOO	0%	5%

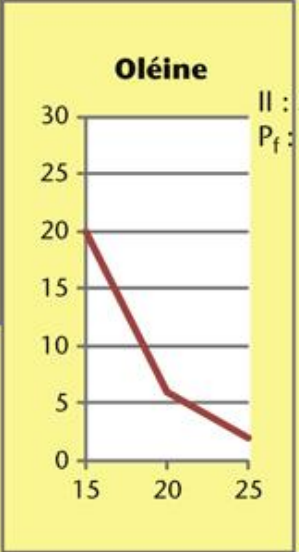
	Oléine	Palme	Stéarine
Indice d'iode	56/58	50/55	32/36
Pt fusion	19/21	36/38	50/55
%S à 15°	18/22	38/42	76/82
%S à 20°	5/7	28/32	67/73
%S à 25°	2	23/27	58/62



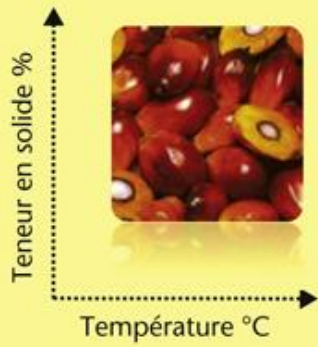
PPP 30 %
PPO 26 %
OOP 44 %
OOO 0 %



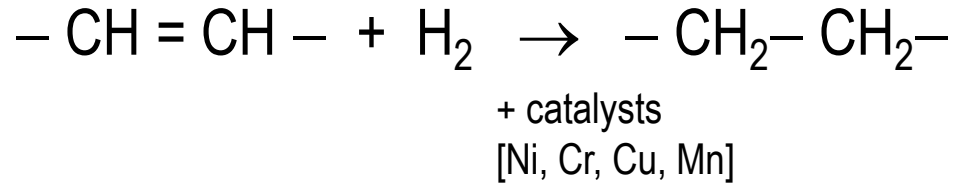
PPP 6 %
PPO 50 %
OOP 40 %
OOO 4 %



PPP 0 %
PPO 51 %
OOP 44 %
OOO 5 %

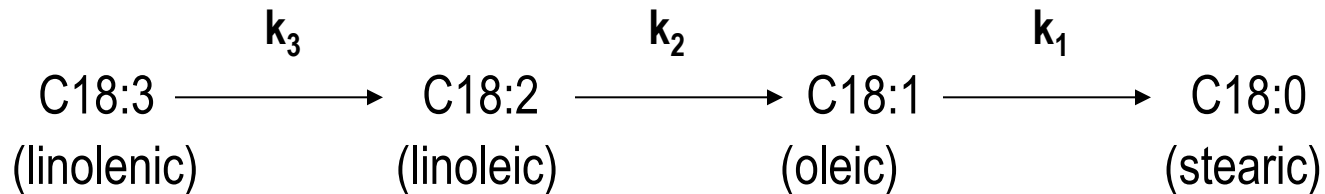


HYDROGENATION



- ↗ melting point
- ↗ stability (rapeseed - soya)

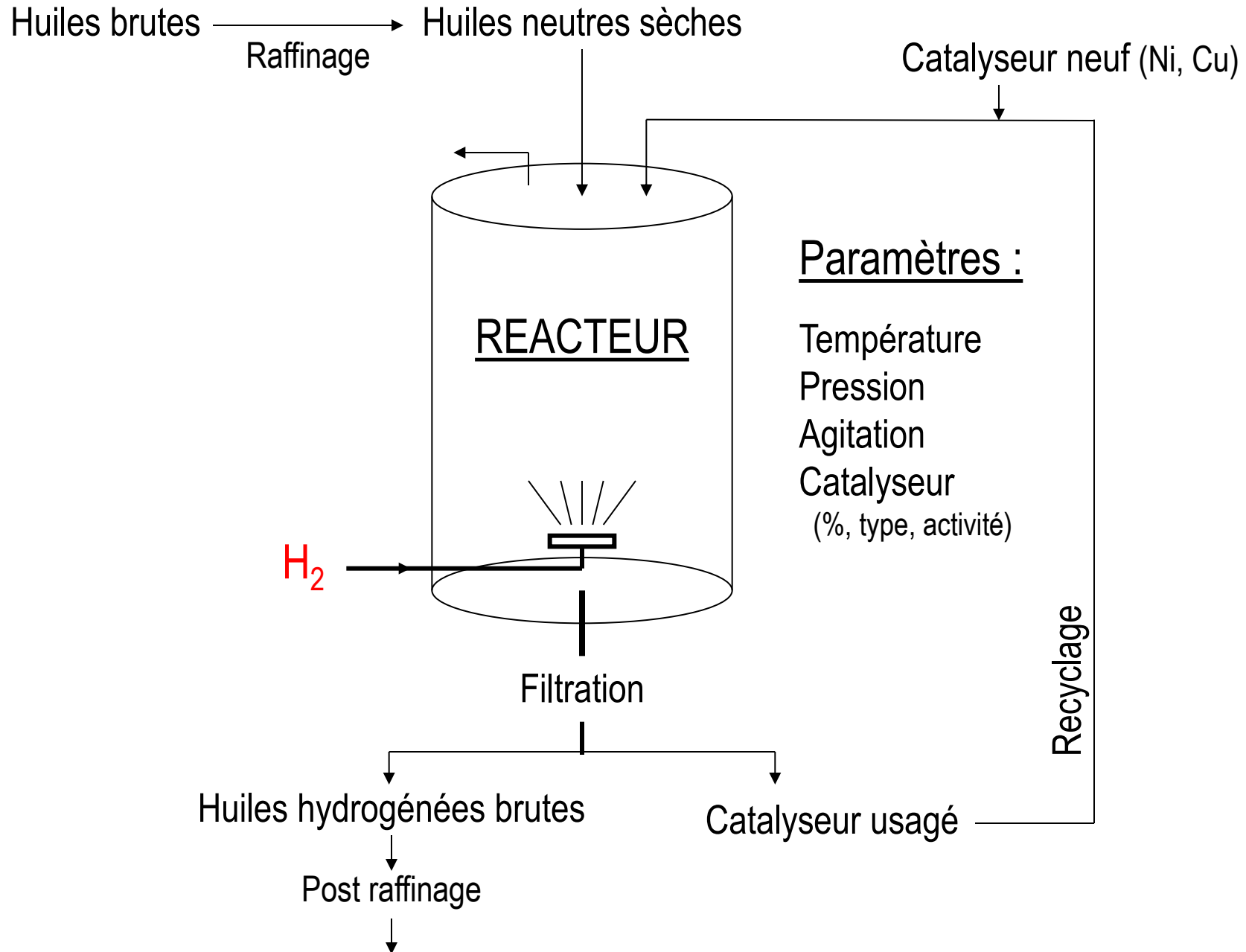
TOTAL or PARTIAL *Hydrogenation, selective or not:*
selectivity = f([catalysts], temperature : k_3/k_2 ; k_2/k_1)



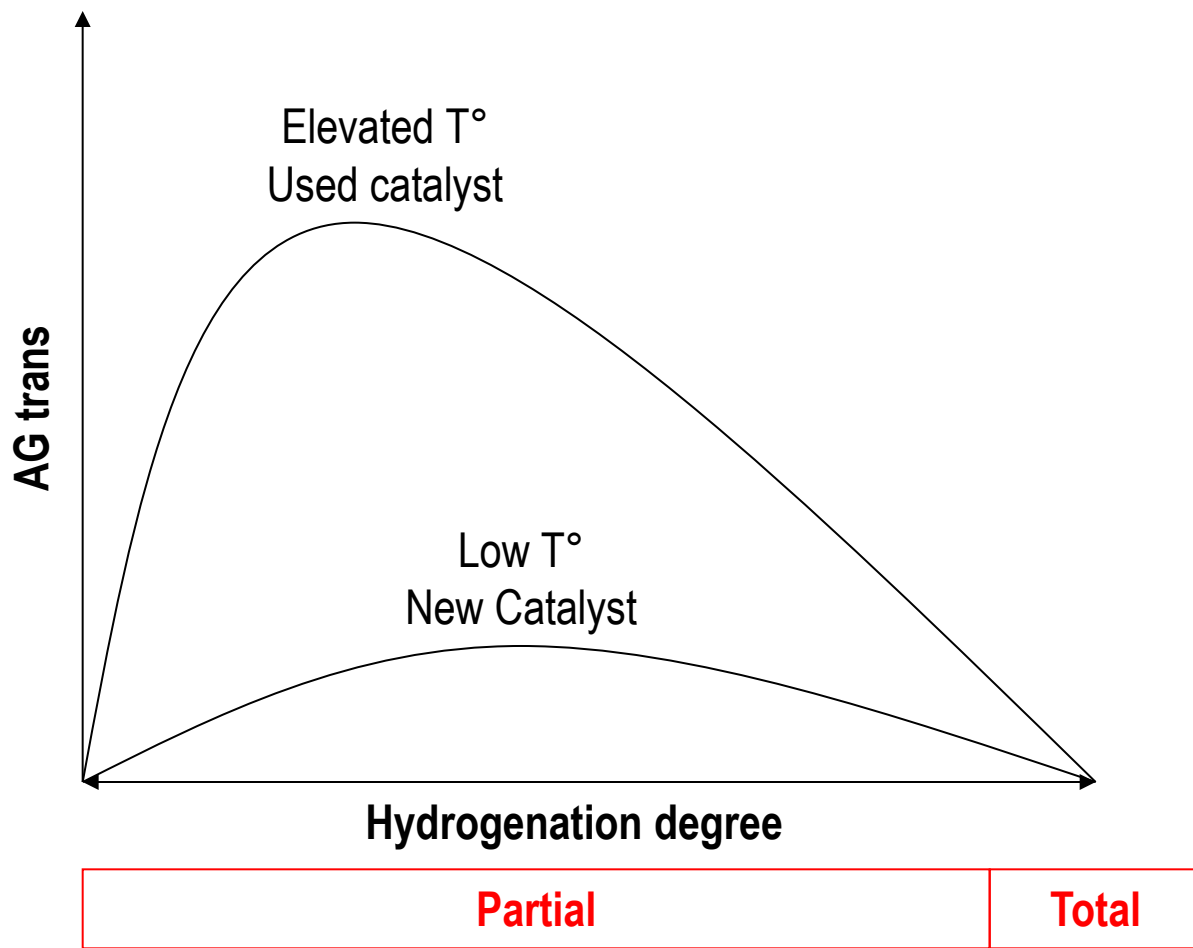
- Apparition of *trans* forms (15 - 40% depending on conditions) ex: elaidic acid (trans- Δ^9 18:1)
- Apparition position isomers (~ 40%), conjugaison (~ 2%)

Applications : raw matter for margarine, pastry, frying oils,

PRINCIPE SCHÉMATIQUE DE L'HYDROGÉNATION



Influence of temperature and quality of catalyst on AG trans formation

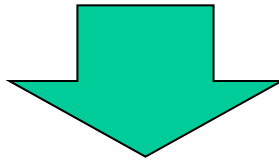


Trans FA content in several food products

AG Trans	Teneur Mini (% AG Totaux)	Teneur Maxi (% AG Totaux)
Sandwich bread	3.7	21.2
Pastry	24.5	34.8
Puff pastry, pizza	16.6	61
Dehydrated soups	4.3	27
Bovine meat	1.0	2.8
Lamb	3.6	5.4
Butter	4	11
Milk (cow/goat/ewe)	1.8	5.6

TRANS FA

- Negative health effects of industrial trans: effect on plasmatic lipoproteines and prostaglandines synthesis
- Compulsory labelling of trans FA



Increase / optimizing of fat processing

- New definition of process parameters (desodorisation, desacidification)
 - lower temperature, shorter time, intense vacuum
- Alternatives to partial hydrogenation :
 - physical fractionation, IE (chimical ou enzymatic) ...



Food



Home > Food

Food	
Recalls, Outbreaks & Emergencies	▼
Foodborne Illness & Contaminants	▼
Ingredients, Packaging & Labeling	▼
Dietary Supplements	▼
Food Defense	▼
Science & Research (Food)	▼
Guidance & Regulation	▼

Trans Fat

This page contains links to all of the content on the Food section of FDA.gov about trans fat. There are links for consumer information, guidance documents, other industry information, science and research reports, and resources from other sites.

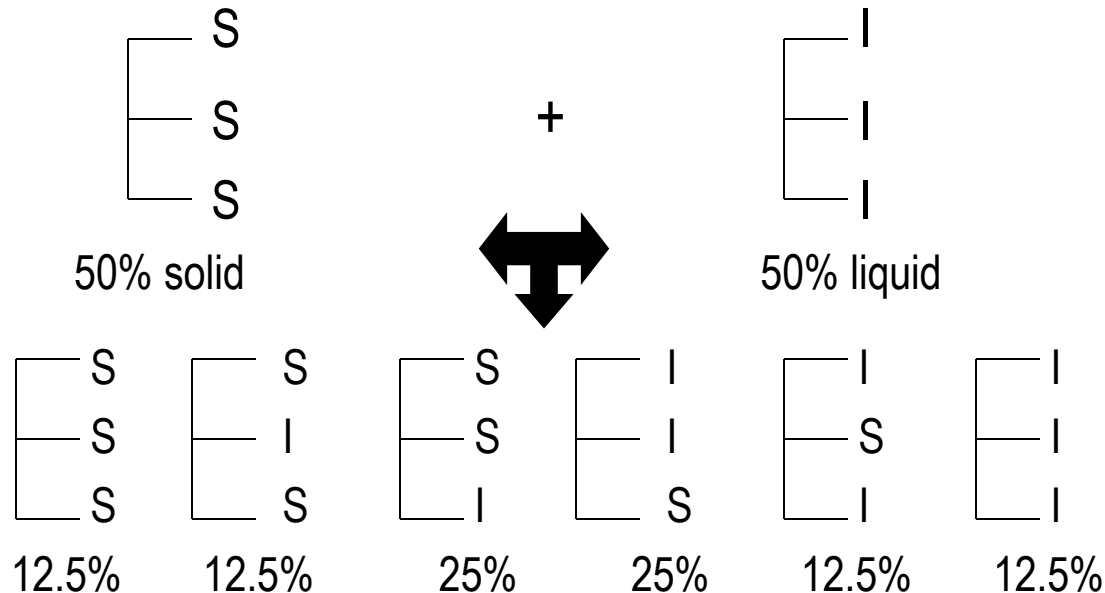
Trans fat is a specific type of fat that is formed when liquid oils are turned into solid fats, such as shortening or stick margarine. During this process — called hydrogenation — hydrogen is added to vegetable oil to increase the shelf life and flavor stability of foods. The result of the process is *trans fat*.

- [Consumer Materials](#)
- [Guidance and Regulations](#)
- [Additional Information for Industry](#)
- [Science and Research](#)
- [Resources from Other Sites](#)

INTERESTERIFICATION

Principle : rearrangement of FA on glycerol

→ modif. Of melting behavior of oil without modification of the oil global FA composition



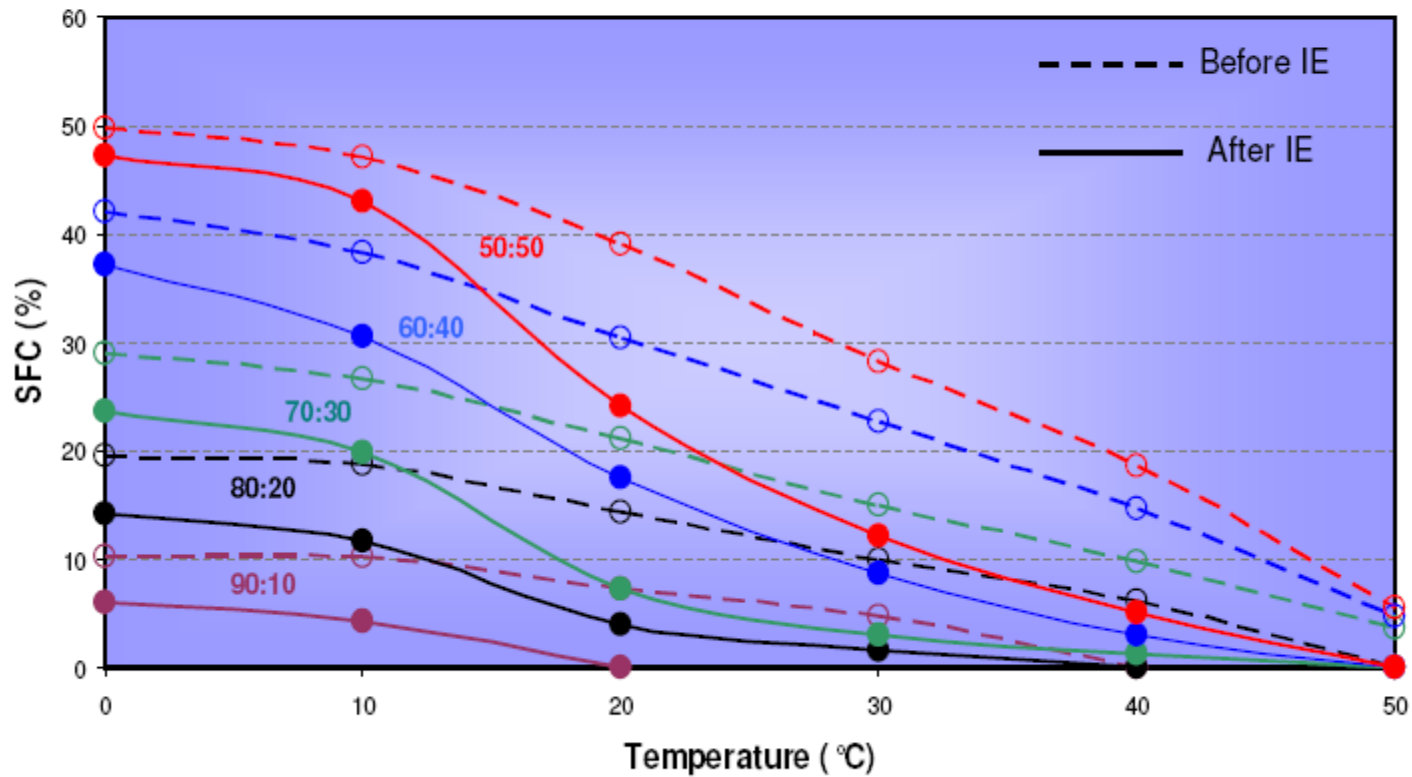
- Non directed : $T > T_{\text{melting}}$ of FA

→ Modification of melting profile

- Directed : T intermediate

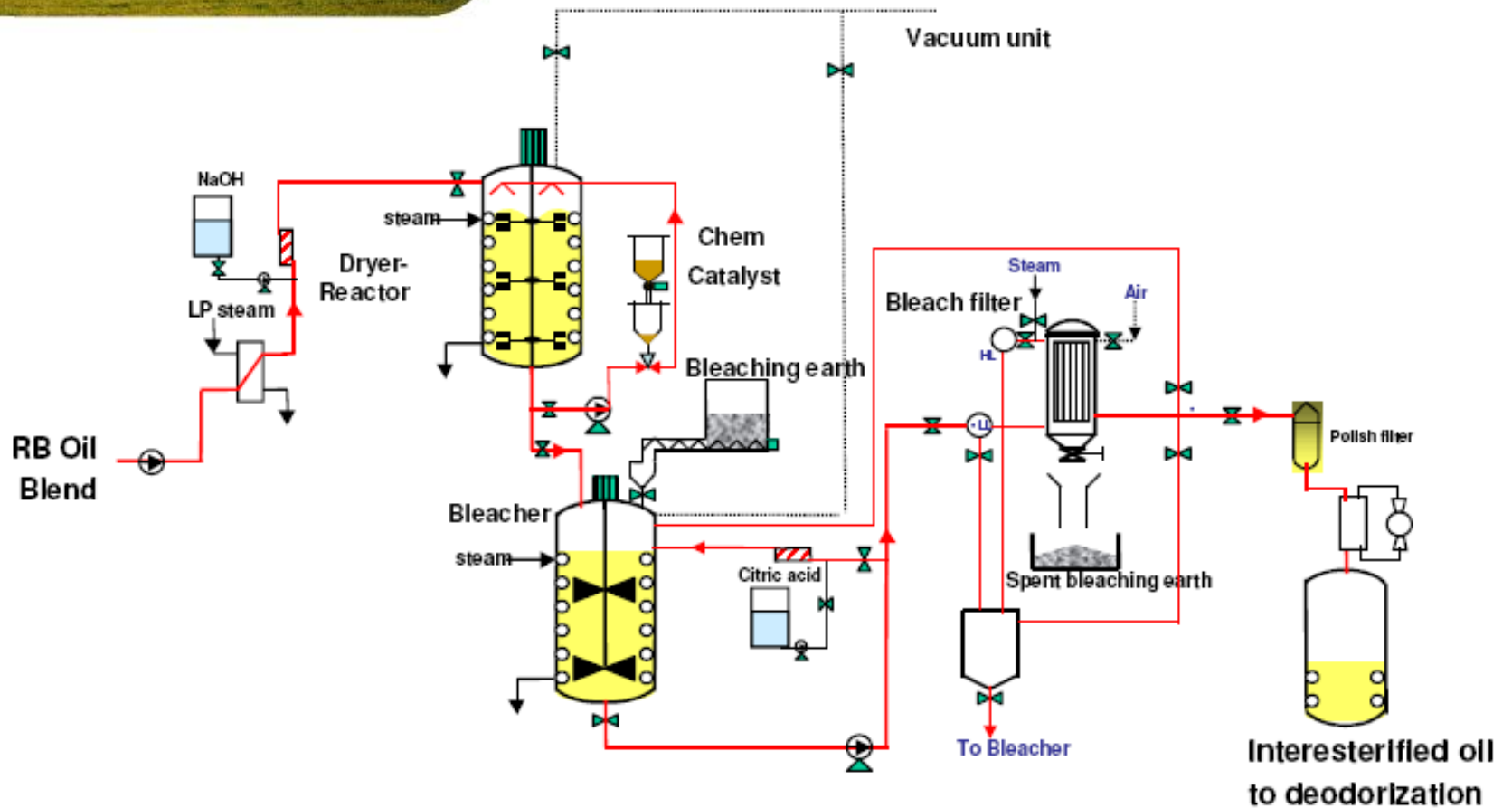
→ Crystallisation of high melting point TAG
(displacement of equilibriums)

Example: Soy Salad Oil + Palm Stearine





CHEMICAL INTERESTERIFICATION



CHEMICAL INTERESTERIFICATION



The Catalyst



Catalyst dry storage tank

Rotary dosing valve

Catalyst dosing tank

Sodium methyate is
a hazardous catalyst



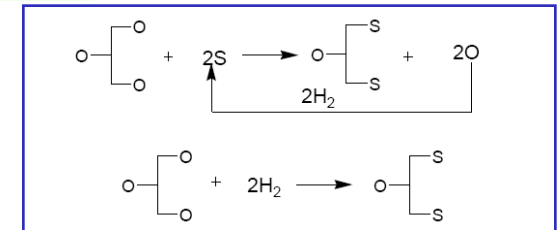
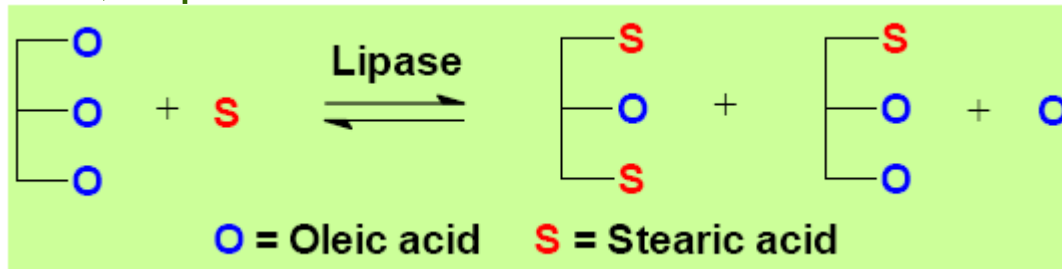
**Special catalyst dosing
systems are important for safe
chemical catalyst handling**

ENZYMATIC INTERESTERIFICATION

Principle : rearrangement of FA on glycerol

→ specificity of biocatalyst to direct modification

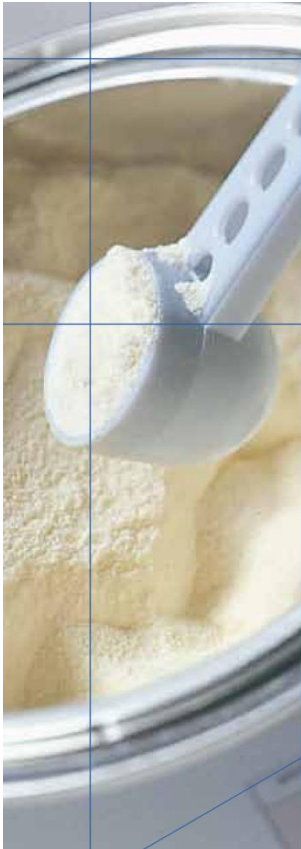
Use of *sn*-1,3 lipases



=> Cocoa butter analogues – texture control

=> Analogue human milk fat in Betapol®, with palmitoyl in *sn*-2 and PUFA in *sn*-1,3





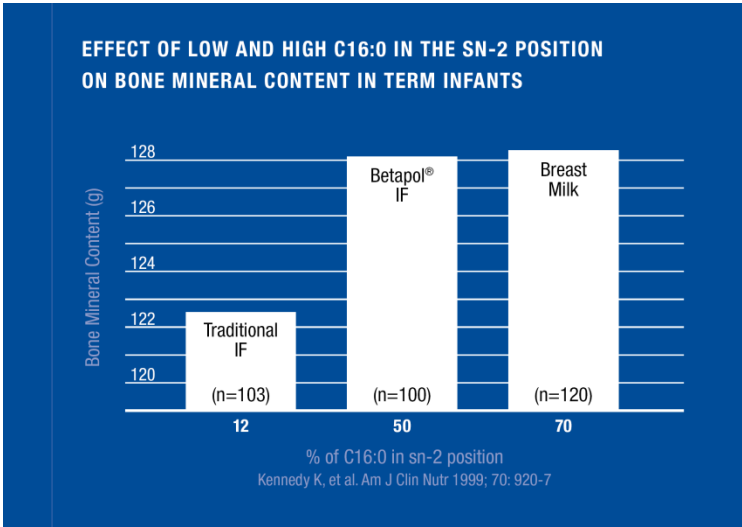
Proven results

The better fat ingredient

Numerous studies with Betapol® conducted in both premature and full-term infants have proven the nutritional superiority of formula blends that have a similar fatty acid composition to human breast milk.

Fat absorption

One leading study compared infant absorption of saturated fatty acids in various control formulas and those that contained Betapol®. All blends had a similar percentage of palmitic acid (C16:0) of 20-25%. In order to mimic human milk fat as closely as possible, the Betapol® formulas were comprised of fats with 66-76% of the C16:0 in the beta position. The control formulas contained 12.6% of the C16:0 in the beta position. Infants that were fed formulas containing Betapol® showed a significant improvement in C16:0 absorption of 22% (pre-term) and 18% (full-term) compared to infants on the control formulas. This clearly established that the more palmitic acid there is at the sn-2 position, the better.



Enzymatic IE (Batch)



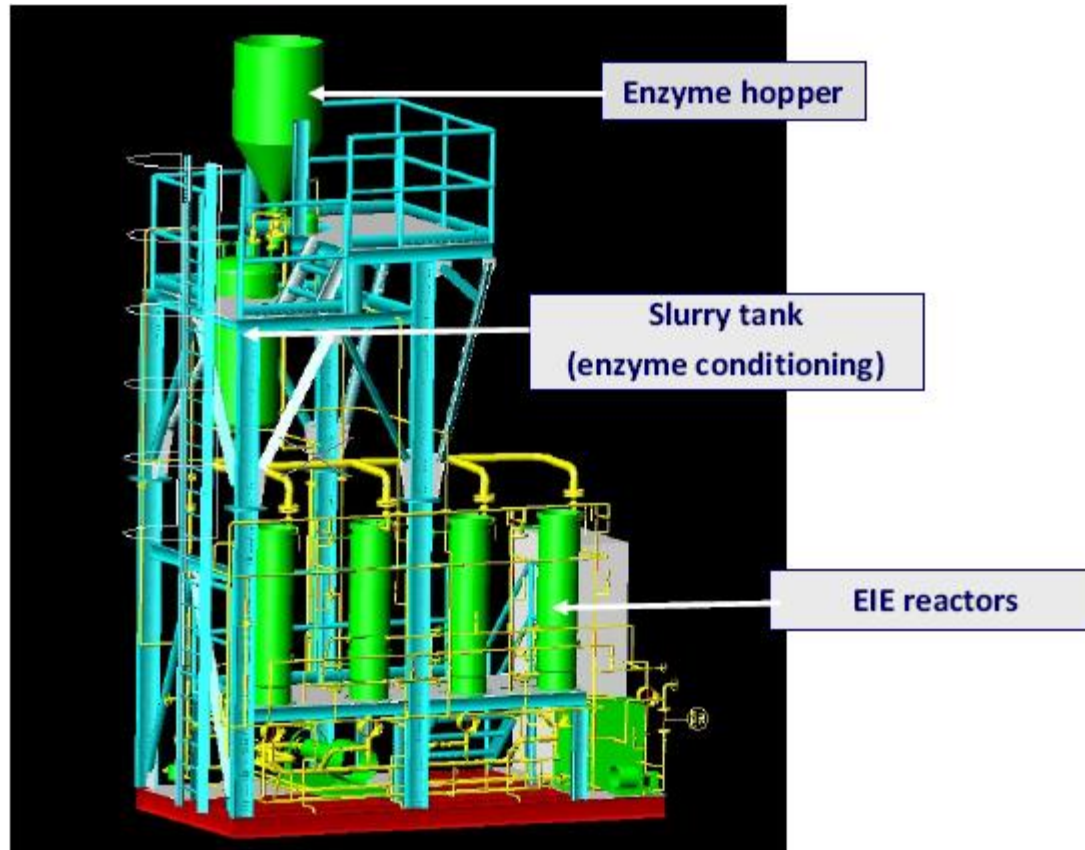
Continuous EIE

Heater
Water bath system
(aquarium)
Columns with enzyme
(+/- 90 g in both)
Pump



Desmet Ballestra



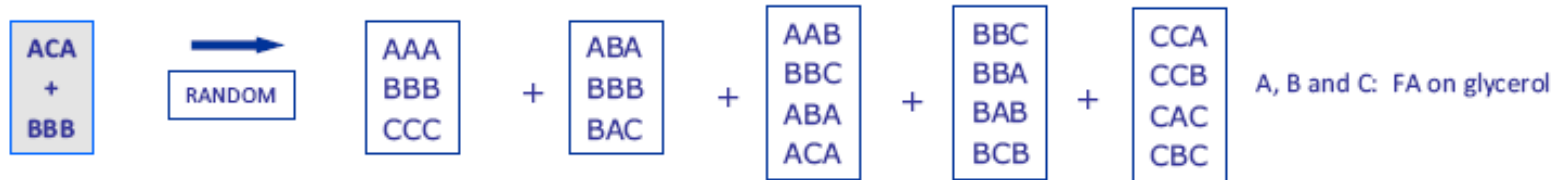


ENZYMATIC INTERESTERIFICATION

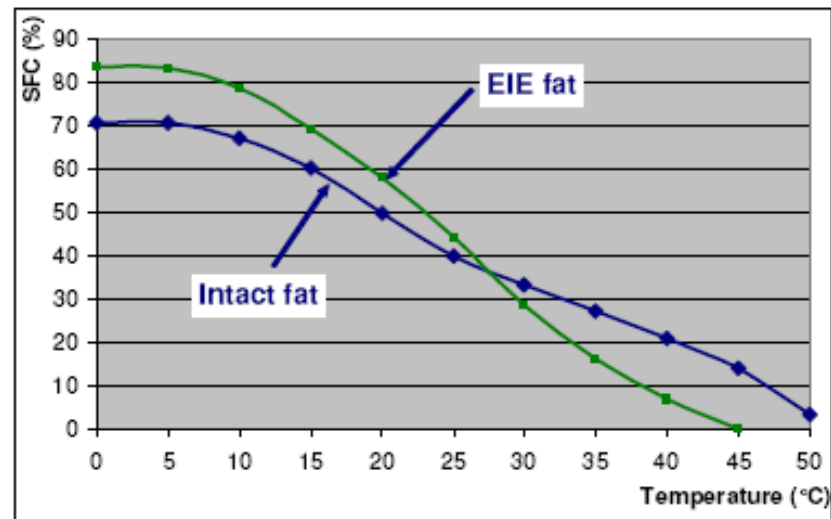
Use of non selective lipases is also possible => randomization

NON-SELECTIVE ENZYME: Lipozyme TL IM, a lipase from *Thermomyces lanuginosus* (Novozymes)

Re-arrangement of FA over the glycerol backbone according to law of probability



Solid fat Content Profile (SFC) by p-NMR



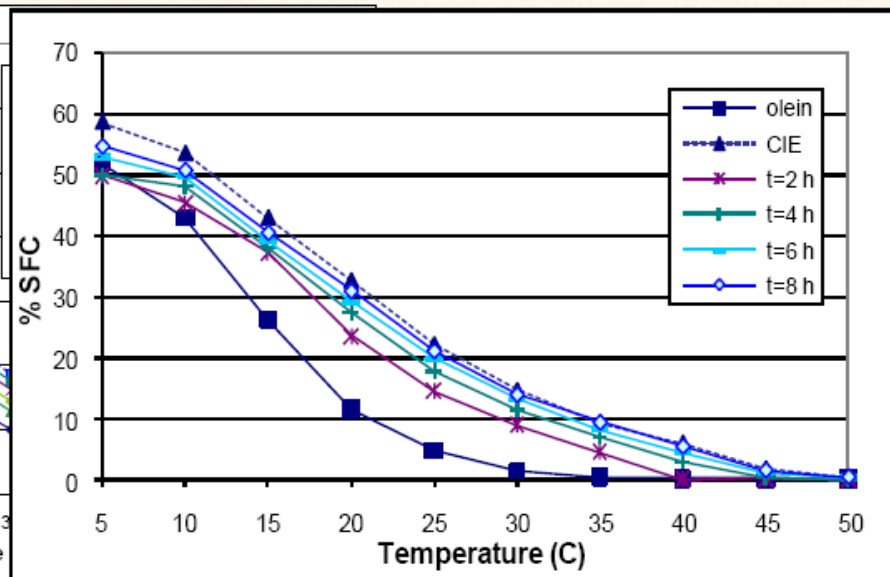
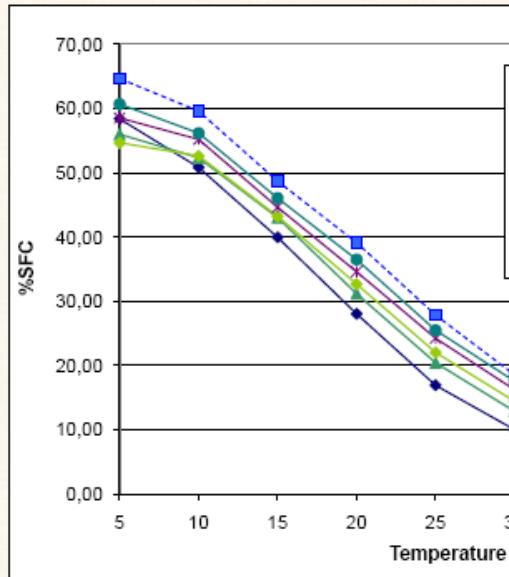
ENZYMATIC INTERESTERIFICATION

Ex application palm oil



Palm oil

Palm olein

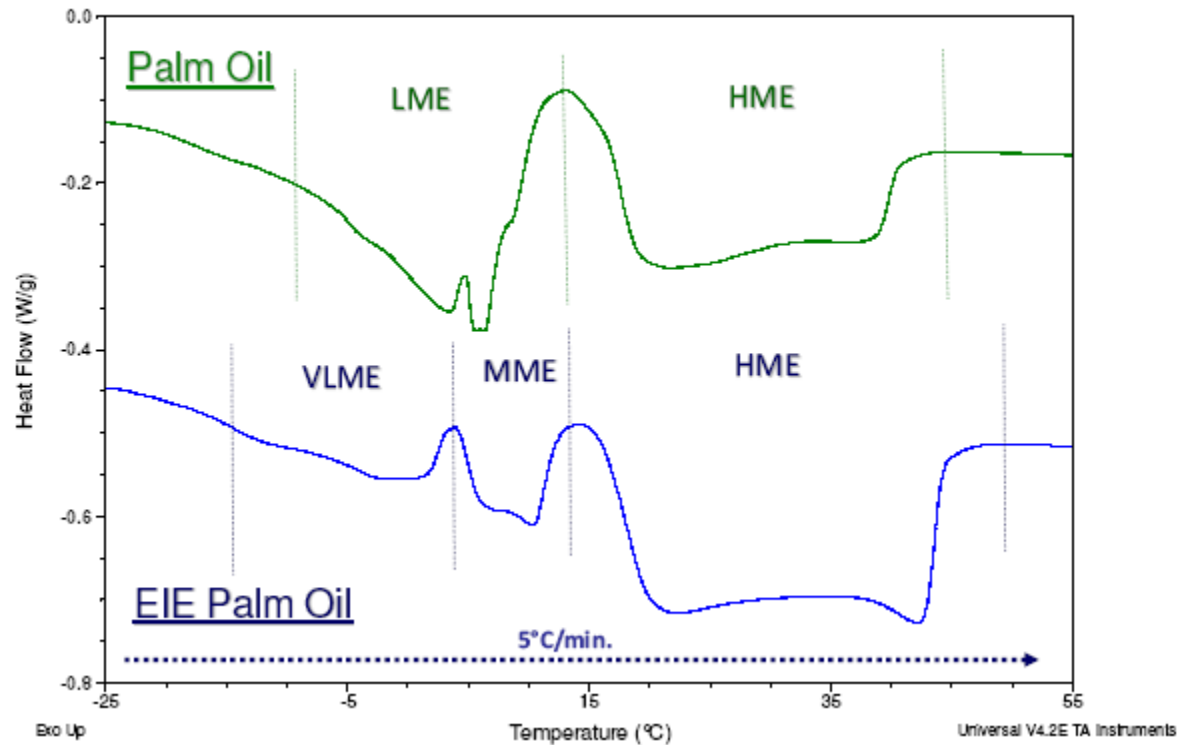


- Increase SFC – eeding with cristals (IV)

ENZYMATIC INTERESTERIFICATION

Ex application palm oil

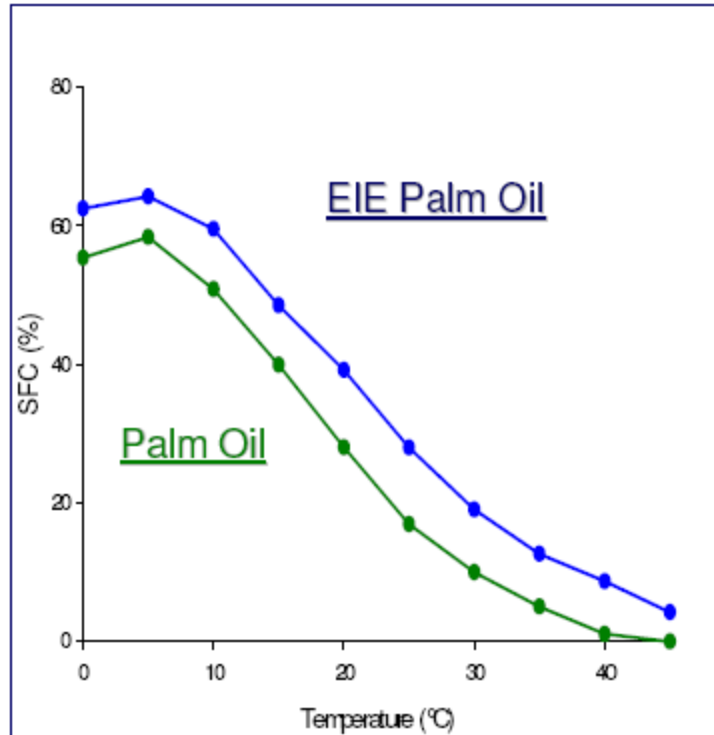
DSC (differential scanning calorimetry) melting profiles of Palm Oil and EIE Palm Oil



ENZYMATIC INTERESTERIFICATION

Ex application palm oil

SFC profiles of Palm Oil and EIE Palm Oil



Composition of Palm Oil and EIE Palm Oil

[St: saturated FA; U: unsaturated FA on TAGs]

Palm Oil

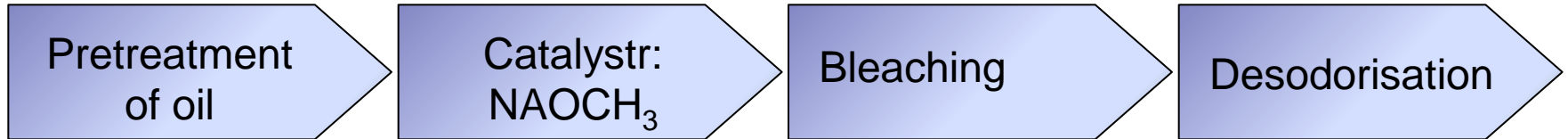
- StStSt: 7%
- StUSt: 46%
- StUU: 40%
- UUU: 7%

EIE Palm Oil

- StStSt: 12% (+++)
- StUSt: 38% (- -)
- StUU : 37% (-)
- UUU: 12% (+++)

PROCESSES COMPARISON

Chemical Interesterification



Enzymatic interesterification



Simpler process but precaution have to be taken to prolonge life of biocatalyst

Random Enzymatic Interesterification



Quality parameters of the feed oil
in order to ensure a sufficient lifetime to the enzyme

FFA: <0.1%

Moisture: >0.02% min.

Moisture & impurities: 0.1% max.

Soaps: 1 ppm max.

P: <3 ppm

Fe: <0.1 ppm

Ni: <0.02 ppm

Cu: <0.01 ppm

AnV: <5

PV: <1 meqO₂/kg

pH of water extract: 6-9

Citric acid: < 25 ppm

Novozymes
Specifications

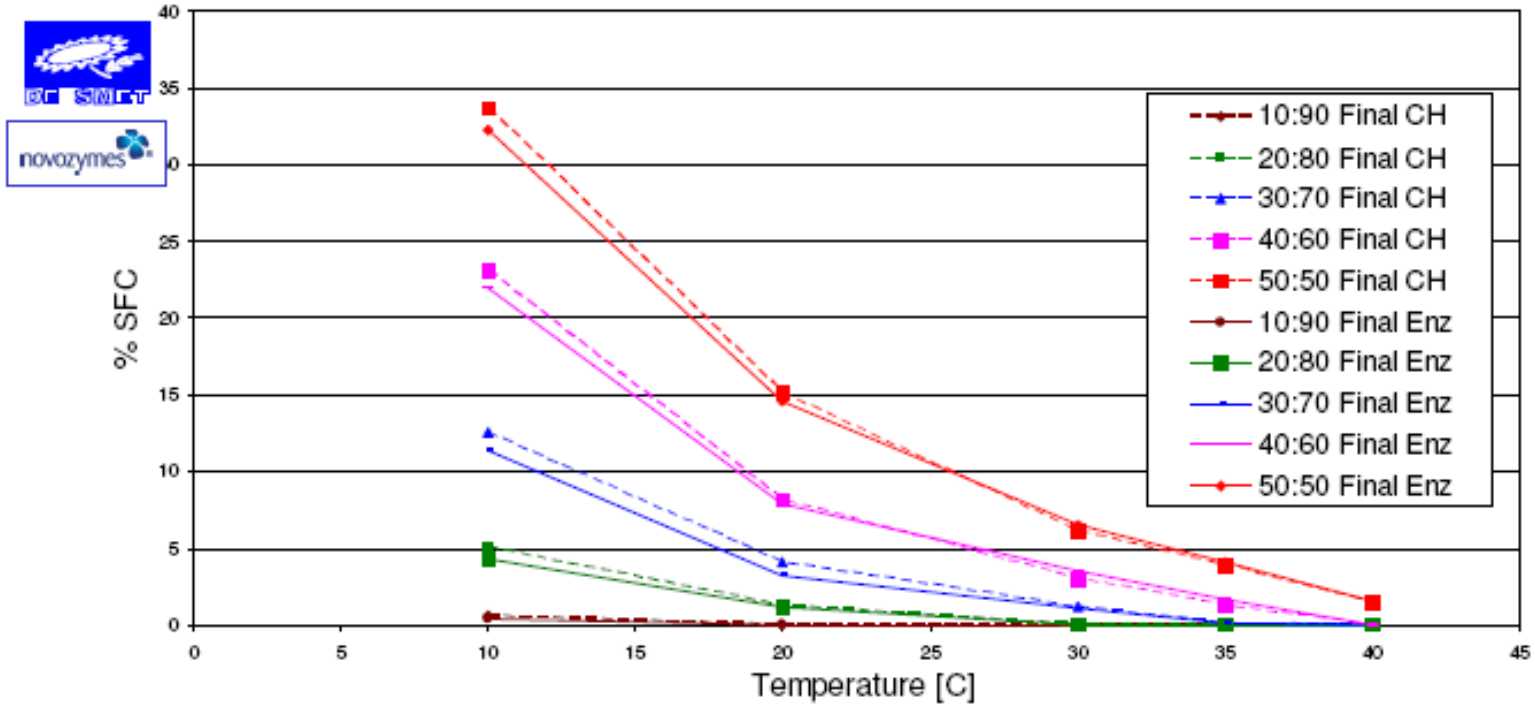
For high, long and consistent enzyme activity, fully refined (RBD) oils are preferred.

High attention with respect to:

- Oxidation parameters, that must be kept as low as possible.
- Acidity of the oil in terms of pH of water extract: > 6 and < 9.

PROCESSES COMPARISON

Soya and palm oils



Sunflower and palm oils

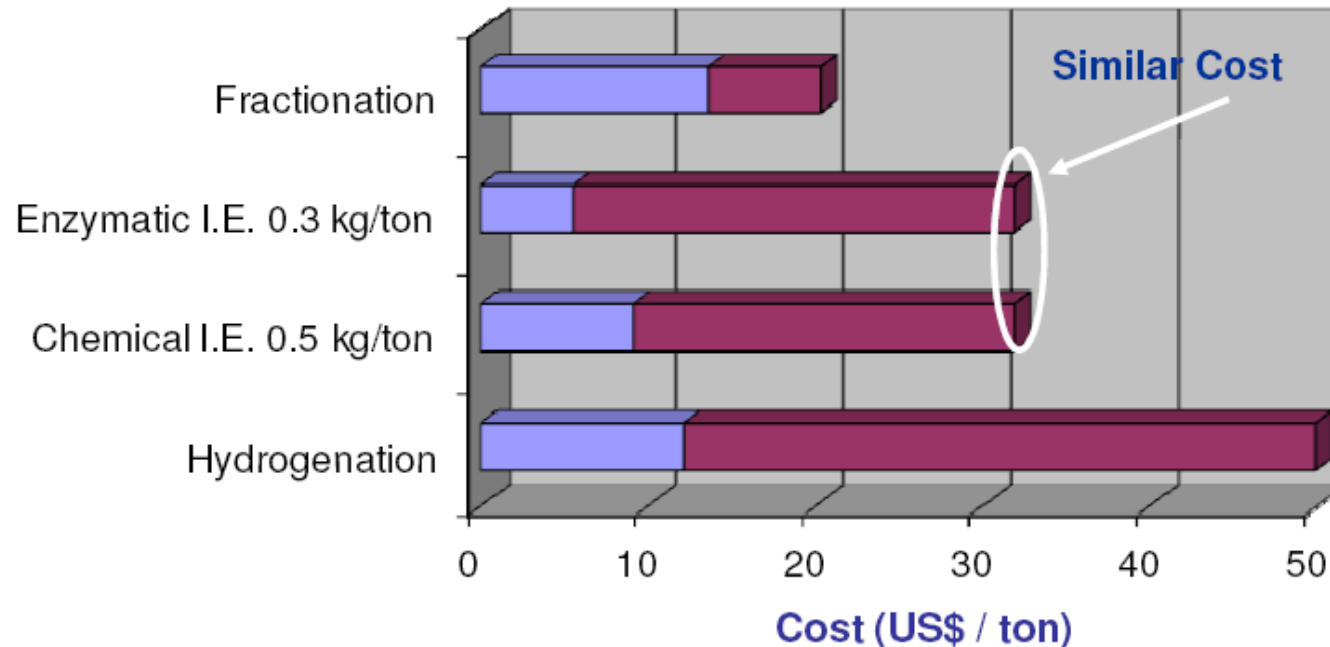
PS / SFO	10/90			20/80			30/70			40/60			50/50		
	Feed	Chem	Enz	Feed	Chem	Enz	Feed	Chem	Enz	Feed	Chem	Enz	Feed	Chem	Enz
Color															
Yellow	11	15	10	15	10	9	16	11	8	19	16	8	19	18	10
Red 51/4	1.0	2.0	1.0	1.2	2.3	0.9	1.8	2.2	1.2	2	3	1.4	2.1	3.4	1.0

Result: Enzymatic process without bleaching makes lighter oil than chemical process after post-bleaching

Tocopherol (ppm)	701	252	505	639	197	412	581	281	426	546	185	425	463	182	366

Result: Enzymatic process retains more tocopherol

Capital and Operating Cost: A Comparison



- 0.5 kg NaOCH_3 randomises 1 ton of oil within a few minutes
- 600 kg enzymes require 1 hour to randomise 1 ton of oil
- NaOCH_3 costs some 4 \$/kg; enzyme costs about 40 \$/kg

PROCESSES COMPARISON

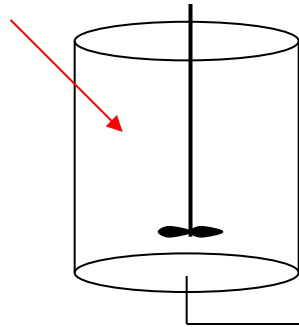
	Chemical IE	Enzymatic IE
advantages	<ul style="list-style-type: none">- Reproducible and efficient- batch operation	<ul style="list-style-type: none">- Reproducible and efficient if no inlet change occurs- simple, clean and safe process- less post-treatment- better nutritional quality of fat- lower initial investment
Drawbacks	<ul style="list-style-type: none">- Toxic catalyst- highTemp- side reactions- off flavours and oxidation- important loss of oil	<ul style="list-style-type: none">- biocatalyst affected by initial purity of oil- hydrolysis- acyls moieties migration- less flexibility

MARGARINE: principle of processing

Fat Phase:

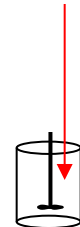
Neutral /bleached oils :

- Fractionated
- Hydrogenated
- Interesterified



Liposoluble Ingredients:

- Lecithine
- Monoglycerides
- dyes



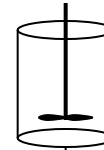
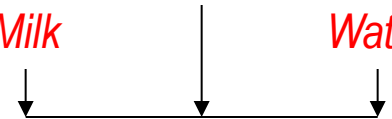
Aqueous Phase:

Additives:

- Salt
- Sugar
- preservative
- Citric acid

Milk

Water



Dosing pump

Emulsion, cooling
Cristallisation, blending

← cold
← Agitation

Packaging

Without trans FA

Refined oil

X

Fraction

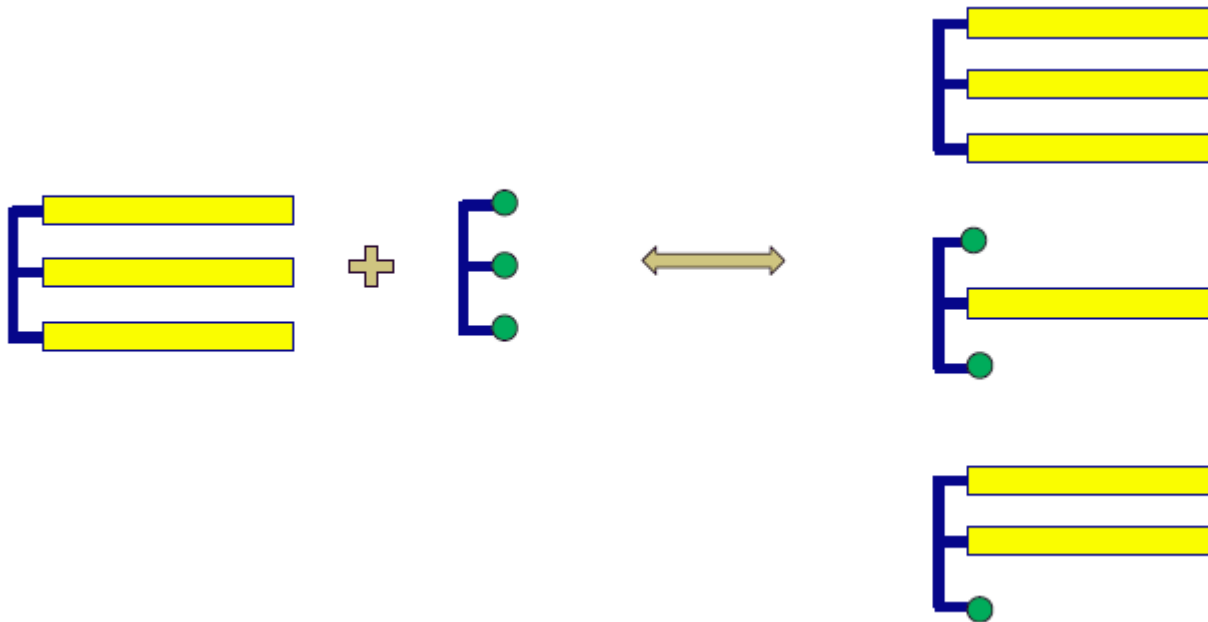
1-X

Fully hydrogenated

Saturated oil

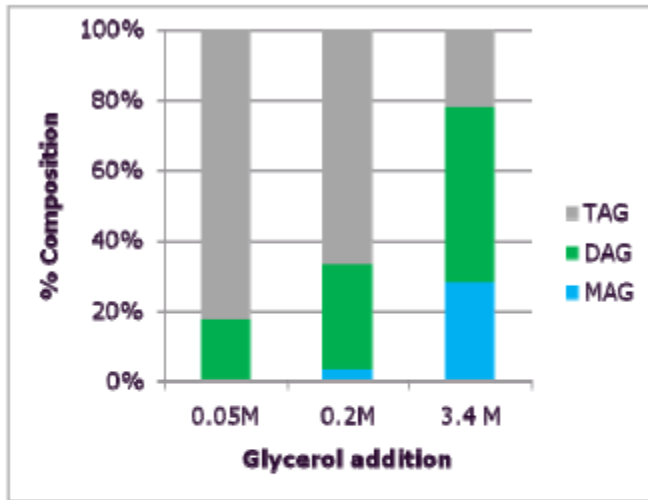
Interesterified

PARTIAL GLYCERIDES PRODUCTION DE BY GLYCEROLYSIS

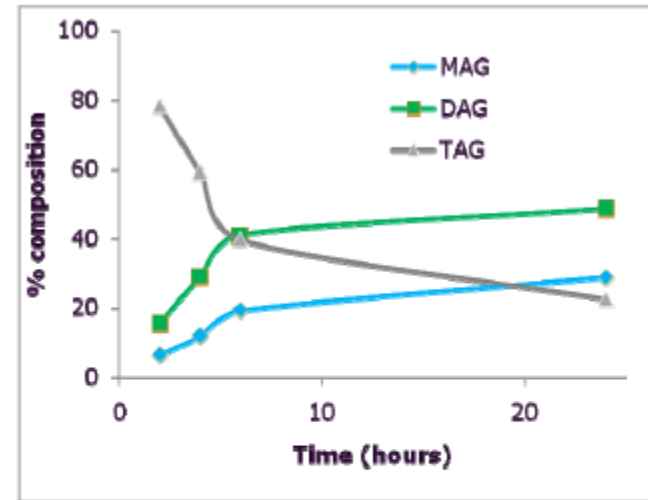


- Used to rearrange FA and produce blends of partial glycerides (MAG, DAG, TAG). Enzyme *sn*-1, 3 or not specific
- Preferred immobilized Enzyme/ elevated content in glycerol

PARTIAL GLYCERIDES PRODUCTION DE BY GLYCEROLYSIS



- Glycerolysis with Lipozyme 435 (2%) and various glycerol addition rates at 80°C/24 hours

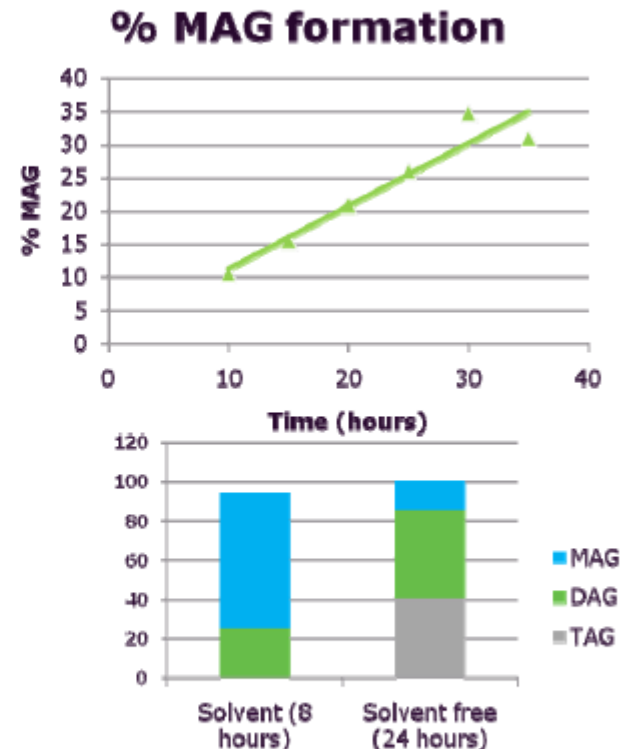


Glycerolysis with Lipozyme 435 (2%) and glycerol (3.4M) at 80°C, 0-24 hours

PARTIAL GLYCERIDES PRODUCTION DE BY GLYCEROLYSIS

Alternative routes

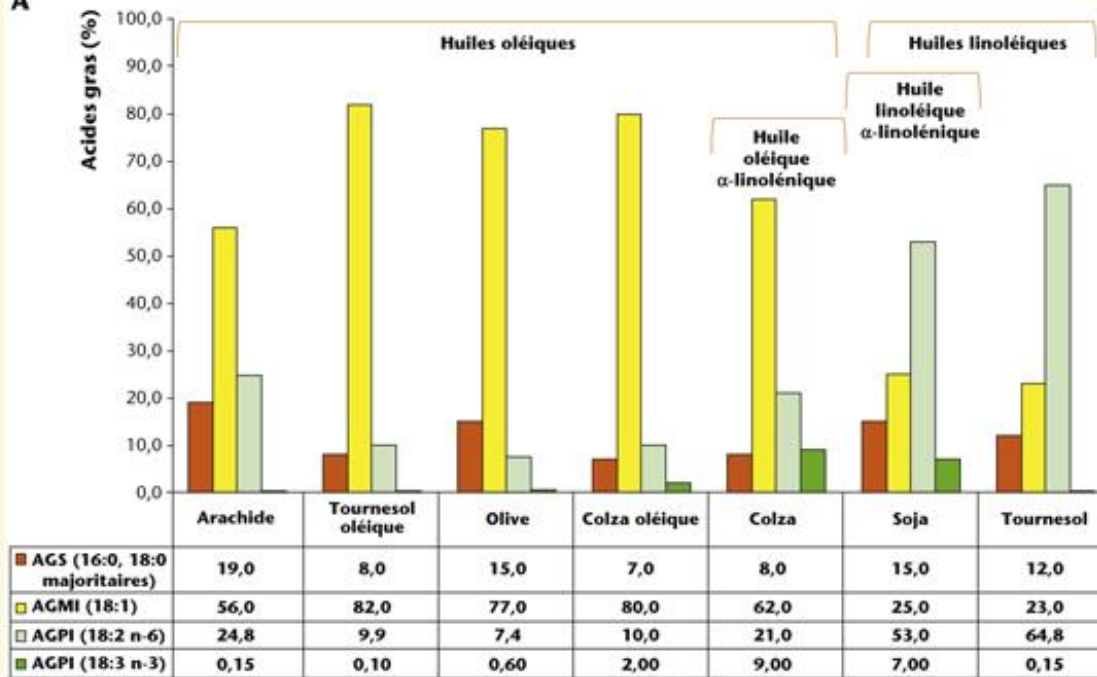
- MAG and DAG can also be synthesised by combining glycerol and fatty acids
- Using liquid enzymes reduces the conversion to DAG but does not eliminate it .
- Presence or absence of a solvent alters the relative proportions of MAG and DAG



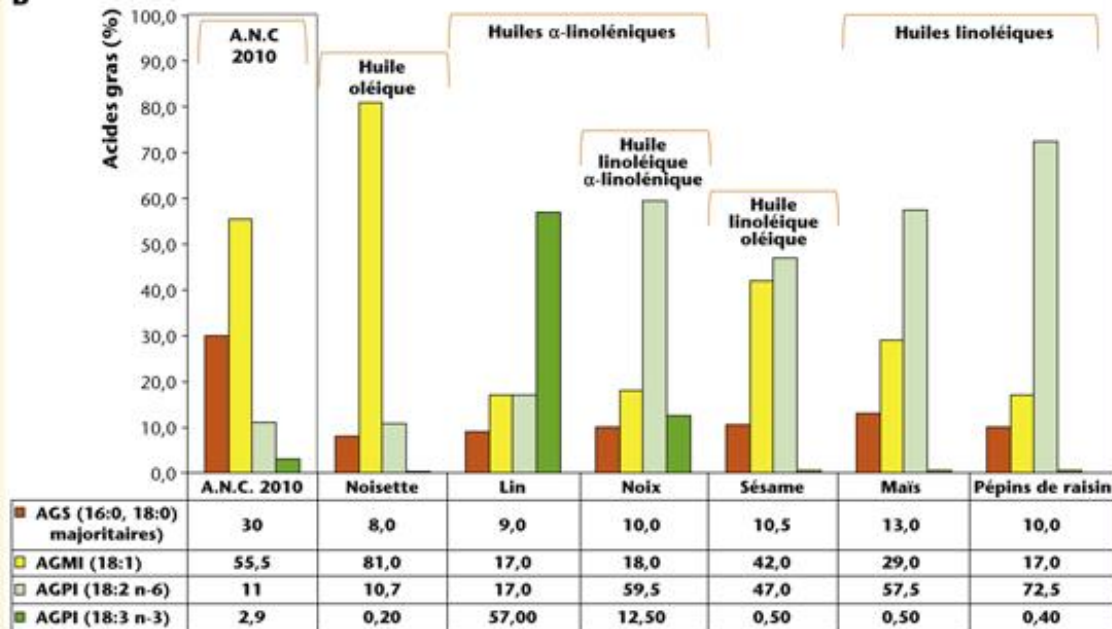
Yang, Rebsdorf, Engelrud, and Xu,
Monoacylglycerol synthesis via enzymatic glycerolysis
in an efficient reaction system,
J. Food Lipids 12, 299-312, 2005.

4. Few oils monographies

A



B



❖ Huiles courantes en alimentation

• Huile de Colza, *Brassica napus*, Brassicacées

Plante herbacée bisannuelle à tiges ramifiées, fleurs jaunes.

Partie utilisée : graines dont on extrait l'huile

Composition : AG oléique, linoléique, palmitique, stéarique.

L'insaponifiable est riche en stérols et tocophérols. Certains sont plus riches en acide érucique que d'autres.



• Huile de Tournesol, *Helianthus annuus*, Asteraceae

Originaire d'Amérique du Nord, cultivé pour ses graines et espèce ornementale. Caractérisé par ses grands capitules dont le réceptacle peut porter 2000 fleurs tubulées, ses grandes feuilles cordiformes.

Partie utilisée : graines dont on extrait l'huile

Composition de l'huile : AG linoléique, oléique, palmitique, stéarique. L'insaponifiable contient de nombreux stérols.



❖ Huile de soja

Source : huile grasse raffinée obtenue à partir de graines de *Glycine soja* et *Glycine max*.

Appartient à la famille des Fabaceae. Culture aux EU, Brésil, Chine, Argentine...

Composition chimique : glucides, protéines contenant les 9 acides aminés essentiels en bonnes proportions, lipides.

Composition en acides gras : linoléique, oléique, linoléinique, palmitique...



Essai :

- détermination des indices habituels
- L'insaponifiable : <1.5%
- CPG pour déterminer la teneur en brassicostérols.

Emploi de l'huile :

- l'huile de soja raffinée est utilisée pour l'alimentation par voie parentérale (apport calorique et d'acides gras essentiels).
- Alimentation.
- **Lécithine de soja** : permet l'obtention de liposomes intéressants pour formuler des émulsions stables. Surtout utilisée dans l'industrie agro-alimentaire (margarinerie et chocolaterie). Contre l'excès de cholestérol et pour la prévention de l'athérosclérose.

❖ Huile d'olive

Source : l'huile d'olive est obtenue à partir de drupes mûres par pression à froid ou par tout autre moyen mécanique approprié.

L'huile raffinée est préparée à partir du fruit par pression à froid, par centrifugation ou par tout autre procédé mécanique reconnu.

Production en zone méditerranéenne.



Drogue :

Fruits (drupe) : alimentation

Feuilles : phytothérapie

Composition chimique : fruit frais riche en eau, en glucides et surtout en lipides.

Composition en AG de l'huile : oléique, linoléique, palmitique,...

L'insaponifiable de l'huile vierge : <1.5%. renferme des stérols, des tocophérols, des triterpènes, des pigments...

❖ Huile de Lin, *Linum usitatissimum*, Linacées

Parties utilisées : Les graines et l'huile tirée des graines.

Habitat et origine : Probablement issue du bassin méditerranéen, cette plante annuelle est aujourd'hui cultivée un peu partout sous les climats tempérés et tropicaux. Elle préfère un sol sablonneux et argileux, voire limoneux, profond et bien irrigué. On récolte la plante après la floraison, avant que les graines ne soient entièrement mûres et ne tombent au sol.



FEW CONCLUSIONS

- 95-98 % lipids = TAG, triester of FA and glycérol
- Hydrolyzed partially before being absorbed by enterocytes , resynthétized under the form of TAG and transported by lipoproteines until target tissues (adipose, liver, muscles...)
- Mutiplr part and essential : energy carrier, structure, signalization, gene expression modulation
- FA : carbon chain with carboxyle moities at one extremity. Chain can be devoid of double bond => saturated FA or present double bonds:

Then FA are said :

- monounsaturated (most frequent in nature oleic acid, MUFA)
- Polyunsaturated (PUFA)

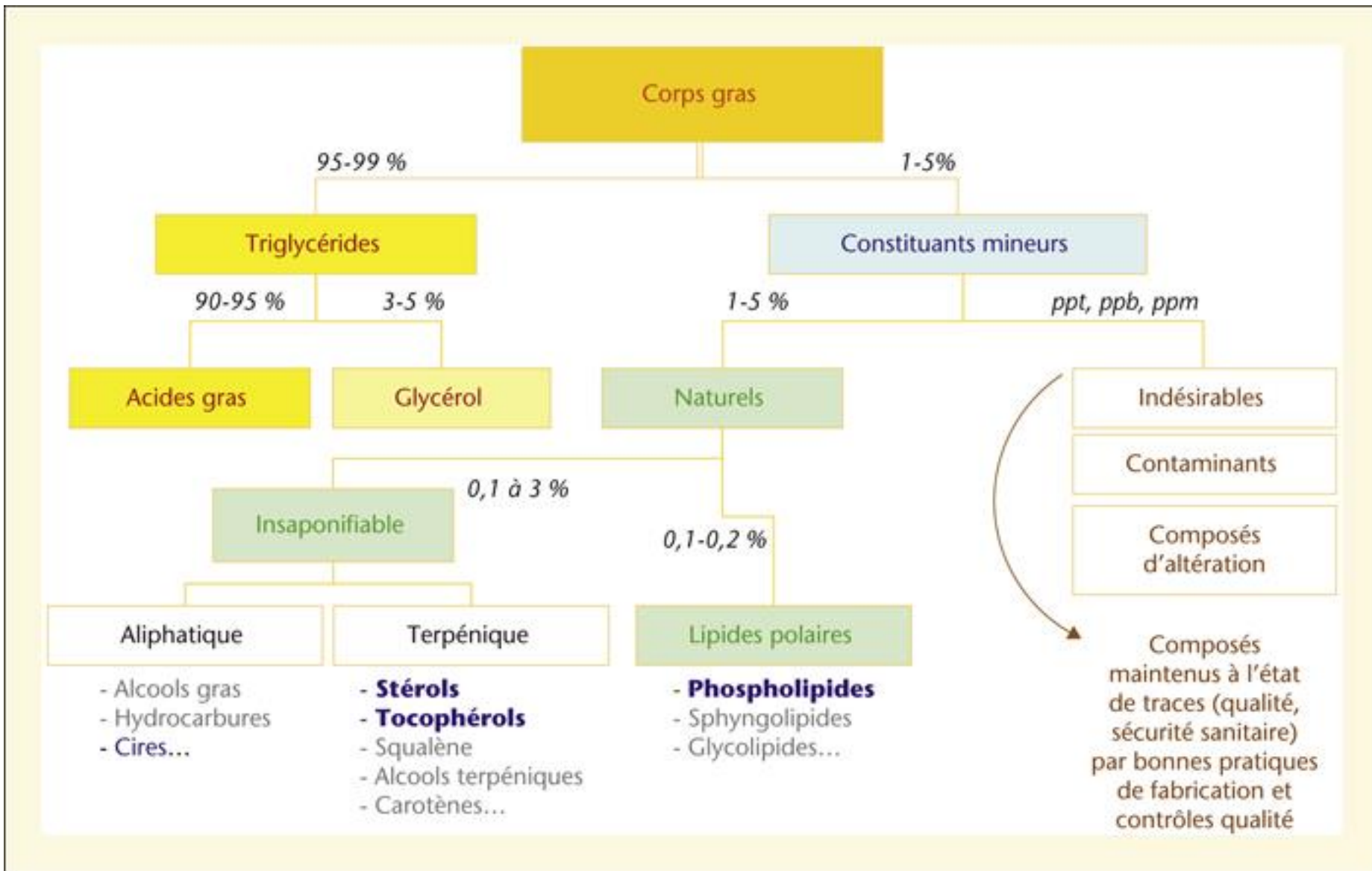
■ 2 series or PUFA very important: n-6 serie (or omega 6) and n-3 serie (or omega 3) with first double bond located respectively at 6 or 3 carbons from the methul extremity of the molecule. Precursor of omega 6 and omega 3 series are linoleic acid (LA, 18:2 n-6) and alpha-linolenic acid (ALA, 18:3 n-3). These two acids are said « essential » because they can not be synthesized by animal organism and must be brought by diet.

■ FA Profile very specific and determine with FA position on TAG => oil properties

■ Chemical and enzymatic process help refig oil and modulated their fonctionnal properties

■ Development of enzymatic method for biomodifications

■ Complex socio economical context: competition for ressources (biofuels), high pressure on agricultural lands linked to emergents markets and increasing demand of ois, complexity of some supply chain (palm), strong image of lipids/fat in collective conscience and complex relationship with health.



Cire de carnauba
La carnaúba est
une cire issue des
feuilles d'un palmier
du nord-est du
Brésil, le Copernicia
prunifera. Elle se
trouve
généralement sous
la forme de copeaux
jaunes-bruns,
cassants, très
odorants



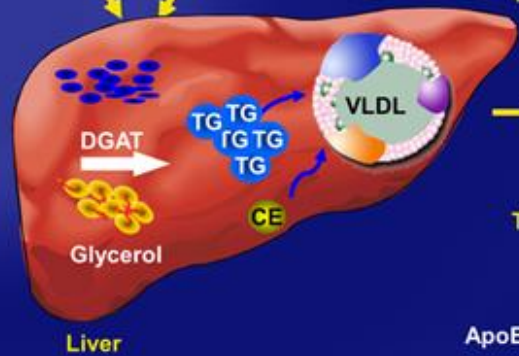
Merci pour votre attention

High TG Levels Impact Other Lipids

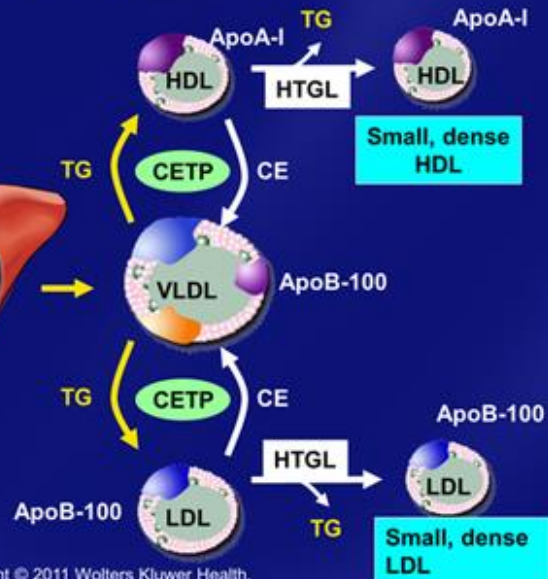


Insulin Resistance

↑ FFA



- Metabolism creates lipid remnants
- Creates small, dense LDL



Miller M, et al. Circulation. 2011;123(20):2292-2333. Copyright © 2011 Wolters Kluwer Health.

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* L. Bellows, Colorado State University Extension food and nutrition specialist and assistant professor; and R. Moore, graduate student. 3/02. Revised 11/12.