UNIVERSITAS PGRI SEMARANG
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PROSES PEMURNIAN MINYAK (2)

(Oil Refining Process)

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In general, fat and oil undergo four processing steps:

1. Extraction
2. Refining (Neutralization or Degumming)
3. Bleaching
4. Deodorization
5. Fractionation

Bleaching

Tujuan utama : untuk menghilangkan zat-zat warna yang ‘tidak’ disukai dalam minyak .

Proses bleaching dilakukan dengan menggunakan suhu kisaran 70-80 ^{o}C) dengan penambahan bahan adsorben tertentu seperti, bleaching clay (earth), arang, atau arang aktif.

Beberapa komponen seperti klorofil dan karoten akan terserap oleh bahan adsorben tersebut.

However, bleaching also promotes lipid oxidation since some natural antioxidants are removed together with the impurities.

Processing Parameters For Bleaching

- Suhu pemanasan harus dijaga pada kisaran70-80 Celsius.
- Proses bleaching dapat dilakukan selama40 – 60 min.
- Dilakukan di dalam tangki yang tekanannya dikurangi
- Jenis dan konsentrasi bahan adsorben sangat mempengaruhi efektivitas proses pemucatan (umumnya ditambahkan sebanyak 0,8 – 1,2% dari berat minyak).

BLEACHING AGENT :

1. Activated Bleaching earth
2. Arang
3. Arang aktif

Activated Bleaching Earth

- Sejenis tanah liat dengan komposisi utama SiO_3, Al_2O_3, air terikat serta ion kalsium, magnesium oksida, dan besi oksida (Ketaren, 2012)
- Daya pemucat bahan ini disebabkan oleh ion Al^{3+} pada permukaan partikel adsorben yang dapat mengadsorbsi partikel zat warna
- Its Catalytic property is self explained by the acid status → aktivasi dengan asam dapat mempertinggi daya pemucat.
- Daya peneyerapan warna akan lebih efektif jika :
  1. Mempunyai bobot jenis yang rendah
  2. Kadar air tinggi
  3. Ukuran partikel halus
  4. pH adsorben mendekati netral

Arang

- Diperoleh dari hasil pembakaran kayu atau bahan yang mengandung unsur karbon (C)
- umumnya memiliki daya adsorbsi yang rendah terhadap zat warna → dapat ditingkatkan dengan cara diaktifkan menggunakan uap atau bahan kimia

Arang Aktif (*Activated Carbon*)

- Aktivasi karbon bertujuan untuk memperbesar luas permukaan arang dengan membuka pori-pori yang tertutup
- Bahan yang dapat digunakan sebagai bahan pengaktif : HNO_3, H_3PO_4, $Ca(OH)_2$, NaOH, Na_2SO_4, $ZnCl_2$, Na_2CO_3, dll
- Can be derived from a variety of coal
- Contain micropores, mesopores & macropores
- It has a large adsorbent surface in a relatively small volume.

Deodorization

- Trace compounds may remain that give a taste and odor to the oil. These are removed by distillation.
- Deodorization can remove some or most of the tocopherol (vitamin E), which is useful for controlling oxidation.
- Deodorizer distillate (residue) is an important source of vitamin E.

Deodorization

Deodorization involves the use of steam distillation under reduced pressure.

In Europe, a deodorization temperature of 175 - 205º C is common, but in the United States, higher temperatures of 235 - 250º C are usually employed.

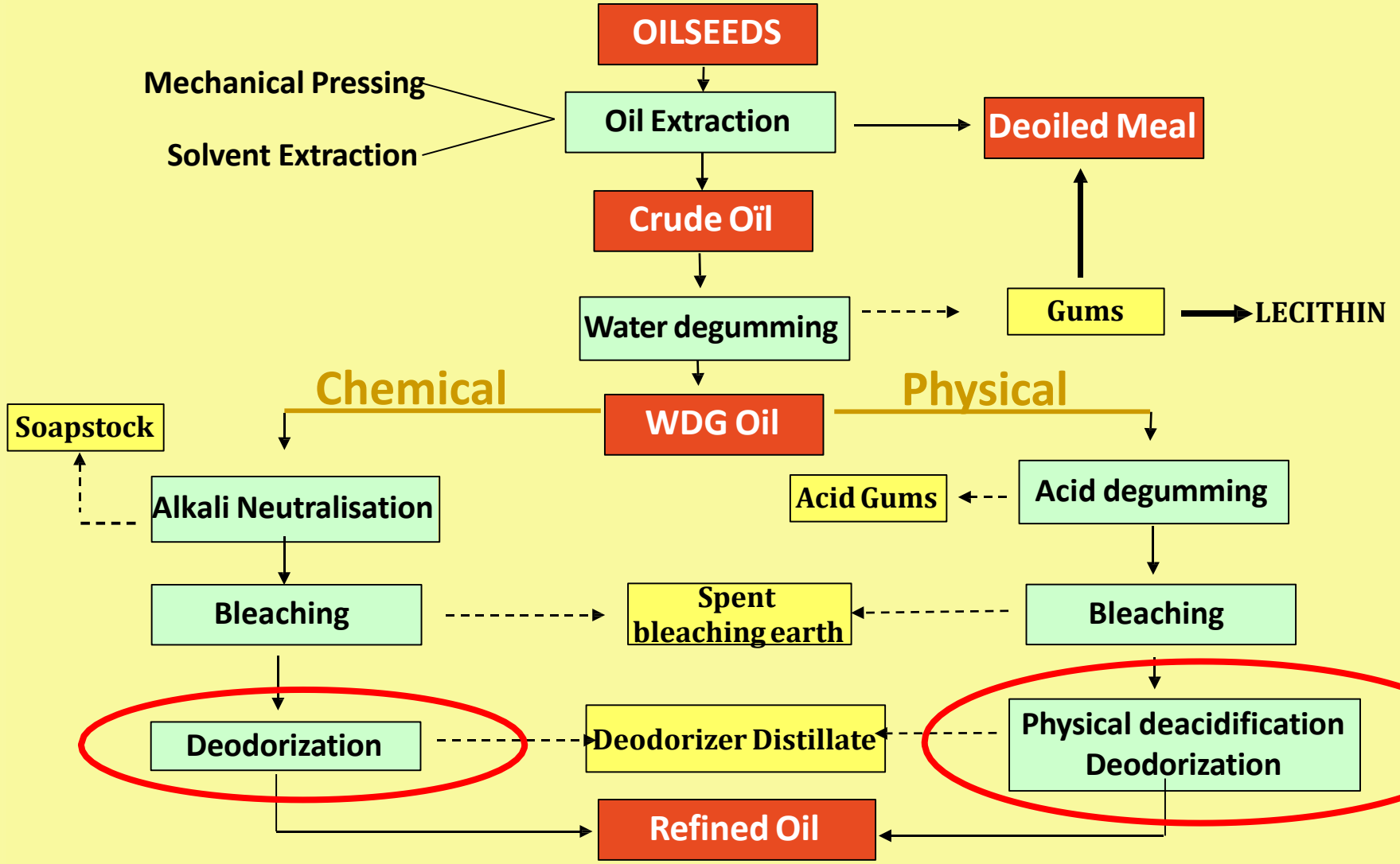
Volatile compounds with undesirable odors and tastes can be driven off, resulting a odorless product.

The oil produced is referred to as "refined oil" and is ready to be consumed or for the manufacture of other products.

About 0.01 percent of citric acid is often added during this step to inactivate pro-oxidant metals such as iron or copper.

Deodorization

Refining (Physical/Chemical)



Deodorization

- Quality defining process in the refining of oils to eliminate the FFA and odoriferous substances.
- Today, the process is still commonly named ‘deodorisation’, but the objectives have become much broader than just the removal of off- flavours.
- High Temperature, High Vacuum, Live Steam Distillation Process
- Distillation is a physical and not a chemical process and does not change the molecular structure of the components

Deodorization : Its Effects

Desirable

Actual deodorisation by removal of different off-flavours

Stripping of volatile components such as:

- FFA (in the case of physical refining),
- Contaminants (pesticides, light, PAH etc.)

Thermal destruction of pigments (so-called heat bleaching)

Undesirable

Some unwanted side-reactions

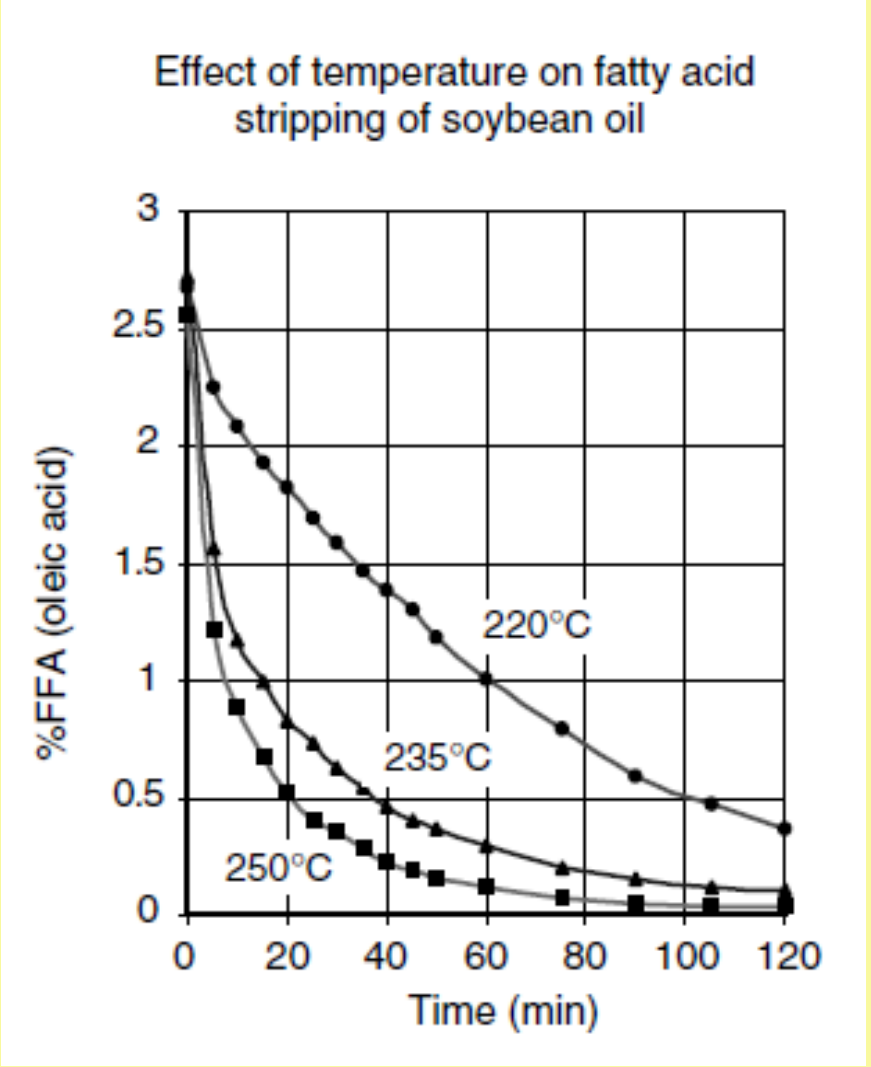
- cis-trans-isomerization,
- polymerization,
- conjugation, and so on

Removal of valuable minor components

- tocopherols,
- sterols etc.

If the oil is not properly pretreated, the oil can become darker during deodorization. This phenomenon is also known as ‘‘color fixation’’

Fats & Oils → (Factors) → Primary Oxidation Products (Hydroperoxides) → (Decomposition) → Secondary Oxidation Products (Aldehydes, Ketones, Diene and triene) → (Oxidation) → Tertiary Oxidation Products (Acids, Epoxides, Dimers, Oxirane rings)

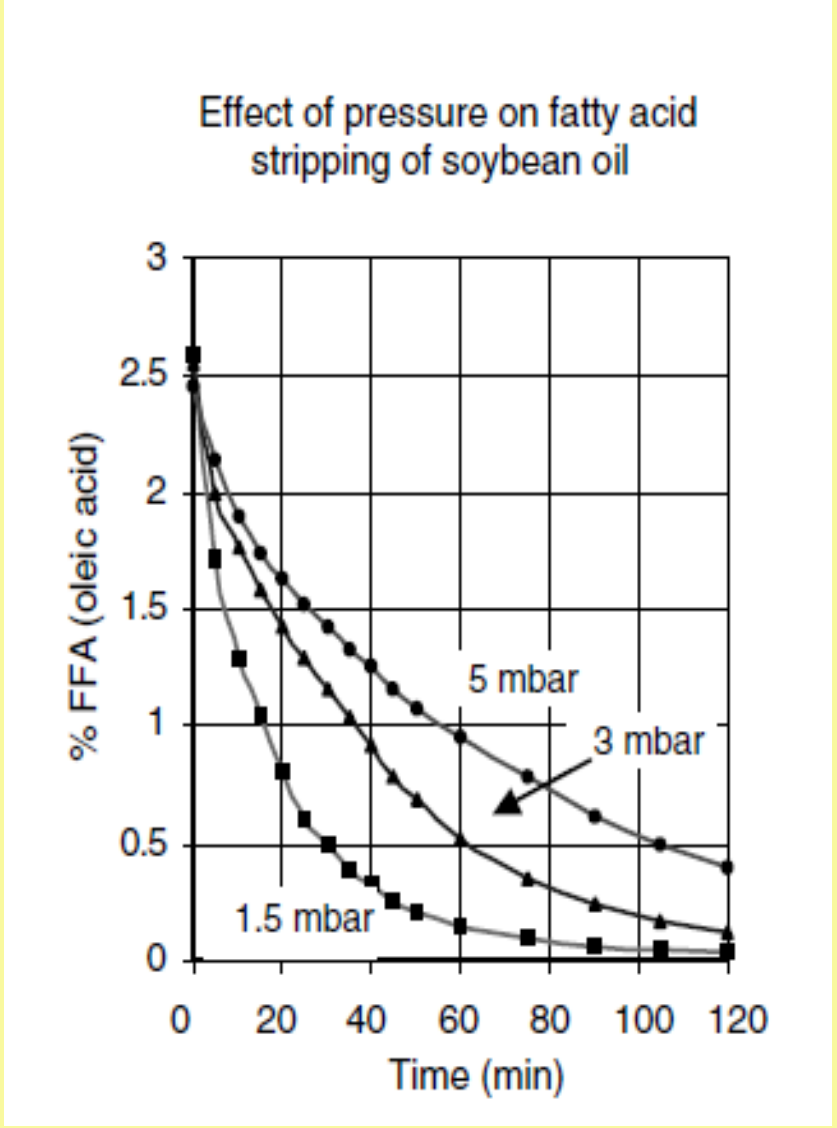


Higher Temperature is avoided because:

- Insufficient thermal stability of oils
- Economy of the process (high energy consumption)

Deodorization : Pressure

- If the pressure is decreased, the temperature required also decreases because a lower vapour pressure is then sufficient to ensure evaporation.
- Reduced pressure helps to protect the oil from oxidation because oxygen from the air is reduced almost to zero.
- The amount of distillation steam required is also directly proportional to the pressure. If the pressure is halved steam consumption also halves.



Higher P means more steam. More steam means more possibility for entrainment. And more stripping steam will increase hydrolysis. This will increase oil losses and lower quality

Stripping Gas

- External pressure of the gas adds to the Vapour pressure of volatiles in deodorizer and speed up their removal.
- Stripping agents with the lowest possible molecular weights are selected. In most cases, steam is the best solution, but sometimes nitrogen is preferred.

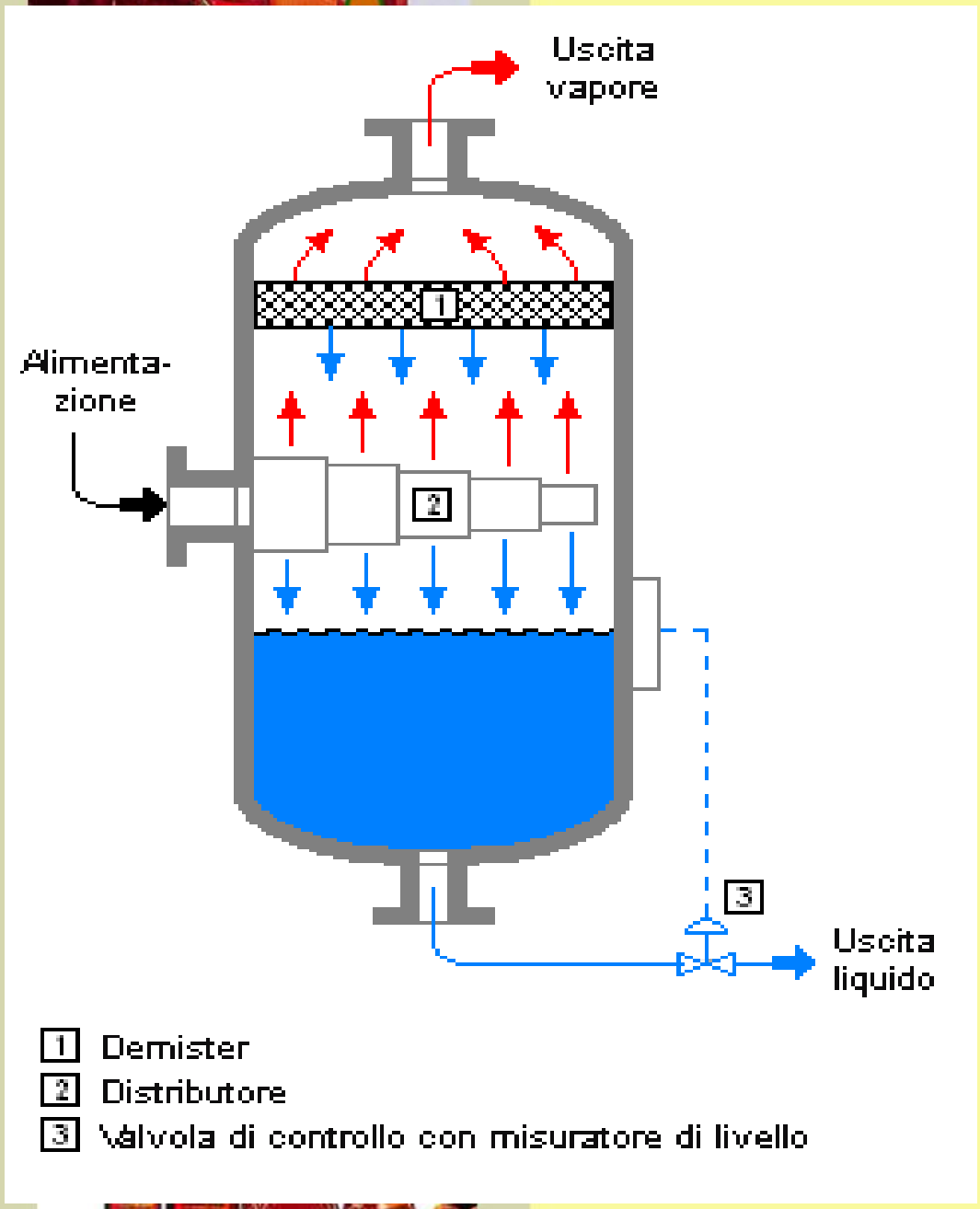
- Since the surface area of the oil is increased by bubble formation, the mass transfer and consequently the distillation rate is increased.

- The height of the oil in the vessel also influences the consumption of steam because the steam has to work against the hydrostatic pressure to be able to penetrate the oil before being sucked off or distilling off from its surface.

It is Conducted at a temperature between **230 and 260 °C**, at a pressure between **2 and 4 mbar** and under injection of **0.5-2% sparging steam**. From a thermodynamic point of view, the stripping agent takes over the part of the total pressure equal to its partial pressure. As a consequence, the vapour-liquid equilibrium is reached at a lower molar fraction, resulting in the removal of significantly more volatile substances only.

- **Nitrogen**
  - inert and non-condensable gas
  - lower losses (no hydrolysis) and higher distillate quality
  - more powerful vacuum system required
  - profitability is very uncertain
- **Steam** (Superheated)
  - most ‘evident’ choice
  - Boost up the vacuum when condensed
  - But support hydrolysis

The sparge gas is introduced into the oil through special steam distributors. These can be sparge coils with very fine holes (between 0.5 and 2.5 mm) or even sintered metal pipes



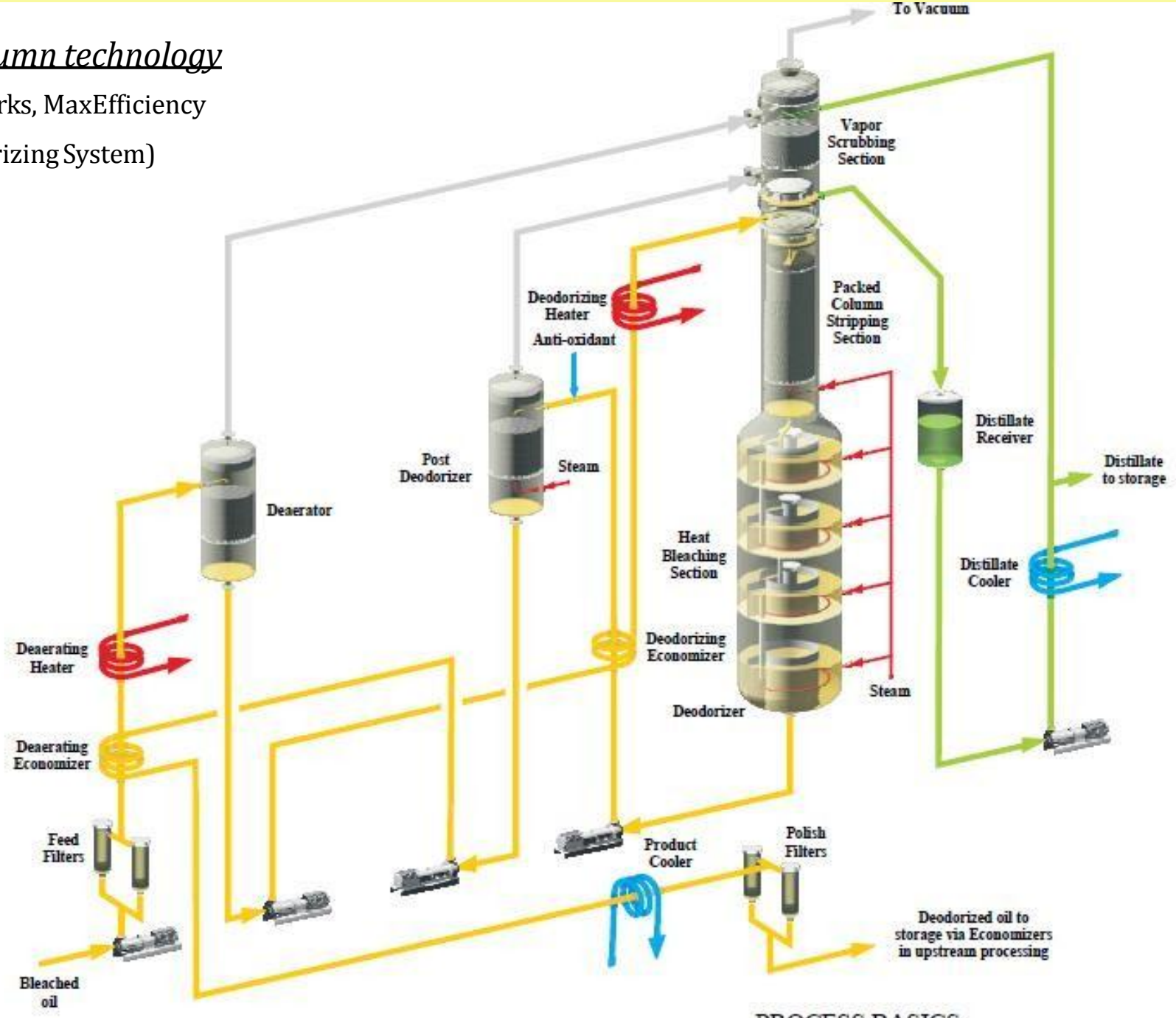
- Composition of vapor phase
  - Volatile components (FFA, odor components)
  - Stripping steam
  - Non condensable gases (air….)

- The volatile substances are condensed by creating an intimate contact between the vapor and fatty acid distillate circulating in the scrubber.

- The distillate which is circulated is at its lower possible temperature (just above its melting point).

Packed column technology

(Crown Works, MaxEfficiency Deodorizing System)



PROCESS BASICS

Effects of Processing Steps on Quality of Soybean Oil

Processing step	Phosphorus (ppm)	Iron (ppm)	Free fatty acids (%)	Peroxide value (meq/kg)	Tocopherols (ppm)
Crude	510	2.9	0.30	2.4	1,670
Degummed	120	0.8	Not determined	10.5	1,579
Neutralized	5	0.6	0.23	8.8	1,546
Bleached	1	0.3	0.08	16.5	1,467
Deodorized	1	0.3	0.00	0.0	1,138

Source: Jung *et al.* (14).

Losses by evaporation

- **FFA** are among the most easily evaporated materials in fats and oils. Normally reduced to below 0.03% by deodorization.
- **Sterols, tocopherols** and other unsaponifiable matter may make up a large portion of the fatty matter are distilled from the oil.
- **Mono- and diglycerides** which have been produced during refining, as they have a lower molecular weight.

Losses by entrainment

- Deodoriser distillate always contains a certain amount of ‘neutral oil’ which has been mechanically entrained from the oil.
- Mechanical entrainment (carryover) of oil by stripping steam is the main unwanted loss in deodorization.

Fractionation:

Fractionation is the removal of solids at a given temperature.

- Crystallization
- Winterization
- Pressing

Crystallization:

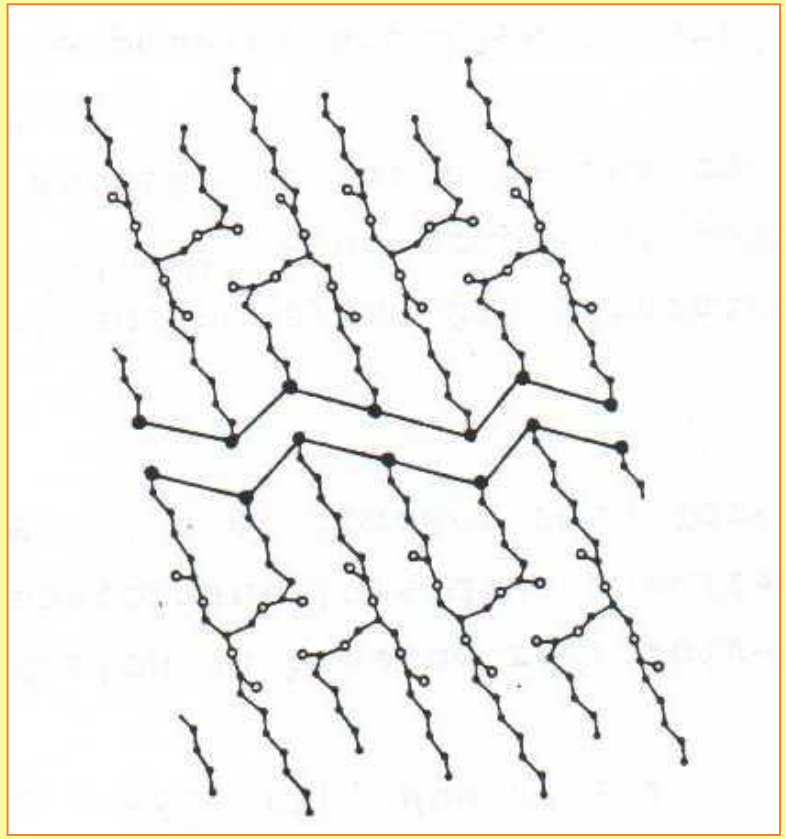
A mixture of trigrycerides is separated into different melting points based on solubility at selected temperature.

Winterization:

Winterization is a process that involves the chilling of the oil to allow the solid portion to crystallize and filtration of the two phases.

RECENT TRENDS IN DRY FRACTIONATION

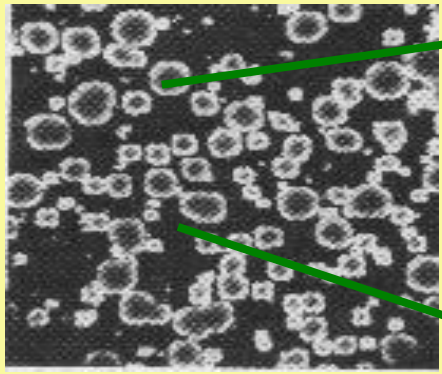
- FRACTIONATION
- DRY FRACTIONATION
- EQUIPMENTS
- WHY FRACTIONATE ?
  - Hydrogenated Soybean Oil
  - Crude Fish Oil (High w - 3)
  - Cottonseed Oil
  - Extra virgin olive Oil
  - Anhydrous Milkfat
  - Beef Tallow
  - Palm Oil
  - Palm Kernel Oil
- CONCLUSIONS



DRY FRACTIONATION

1) CRYSTALLISATION :

CONTROLLED CRYSTALLISATION OF THE MELTED OIL, CONDUCTED ACCORDING TO A SPECIFIC COOLINGPROGRAM.



STEARIN :
Solid fraction

OLEIN :
Liquid fraction

2) SEPARATION OF THE CRYSTALLISED OIL :

Vacuum belt (or drum) filter : Soft stearin.

Membrane filter press : Hard stearin. Higher yield in olein.

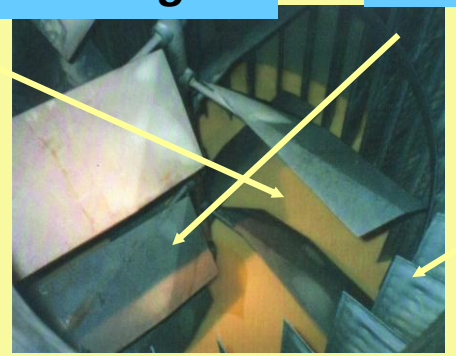
Nozzle centrifuge : Soft stearin. Closed and small space.

EQUIPMENT : CRYSTALLYSERS

Type 1 :

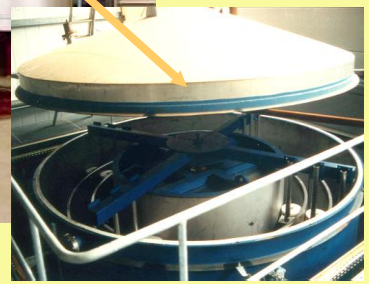
Crystallizing oil

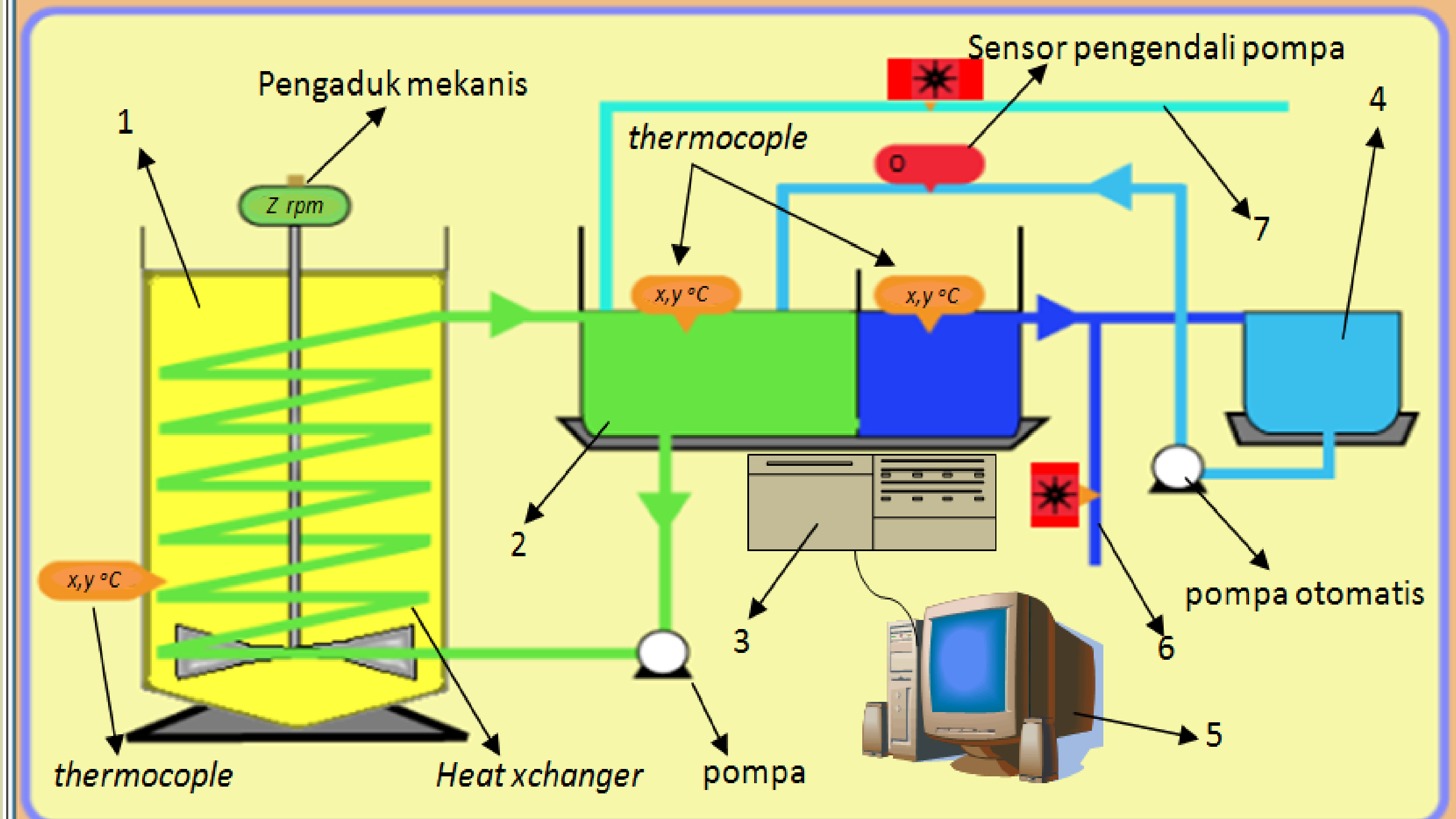
Agitator

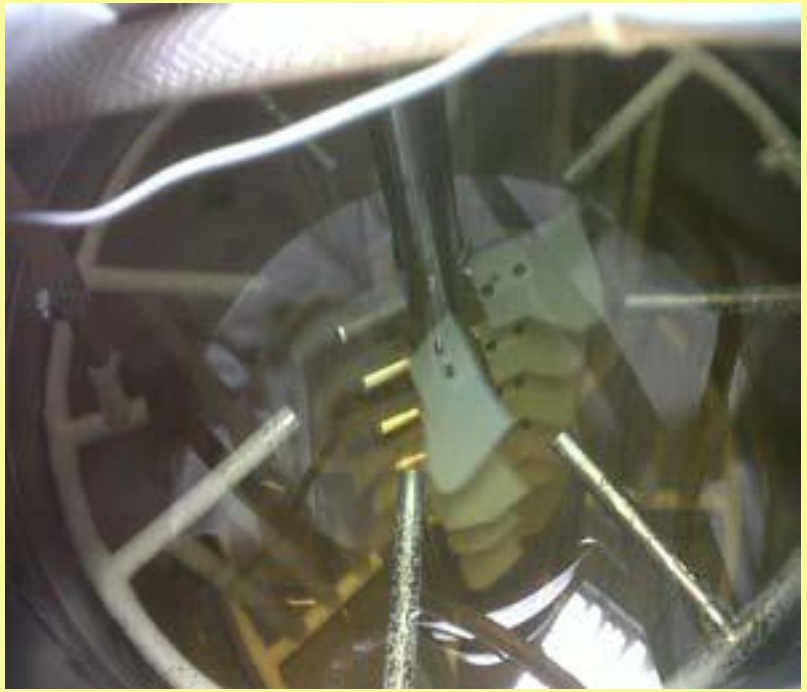


Exchange plates

Type 2 :




Sensor pengendali pompa
Pengaduk mekanis
1
thermocople
4
Z rpm
0
7
x,y °C
x,y °C
2
x,y °C
3
6
pompa otomatis
thermocople
Heat xchanger
pompa
5

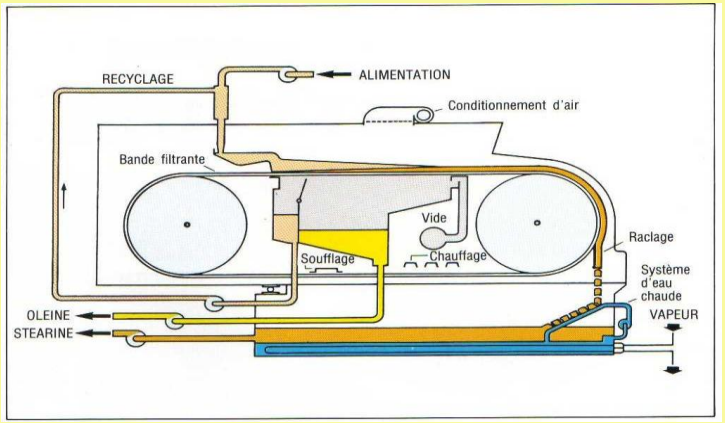


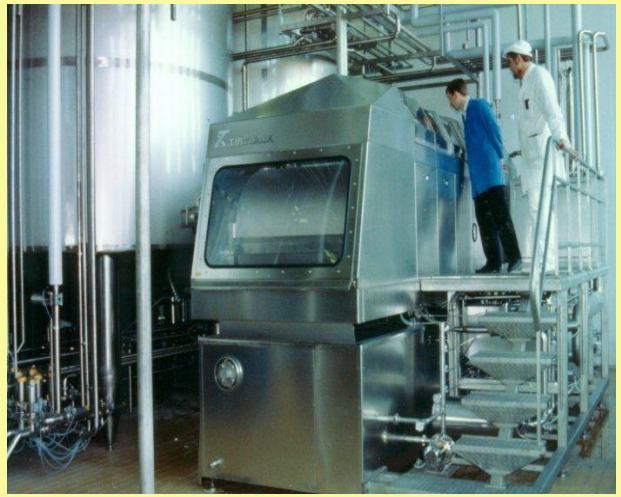

1 2 3 4 5 6

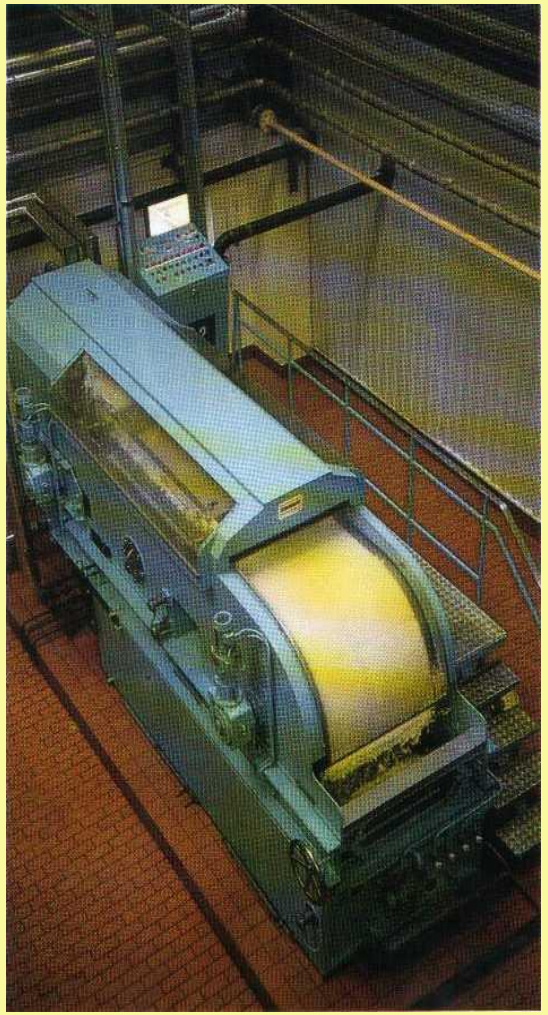

DAISY
NATURE

EQUIPMENT : SEPARATION

Florentine filter :







Scraping of the stearin cake

EQUIPMENT : SEPARATION

Hermetic membrane filter press :

Discharge of the stearin cakes