

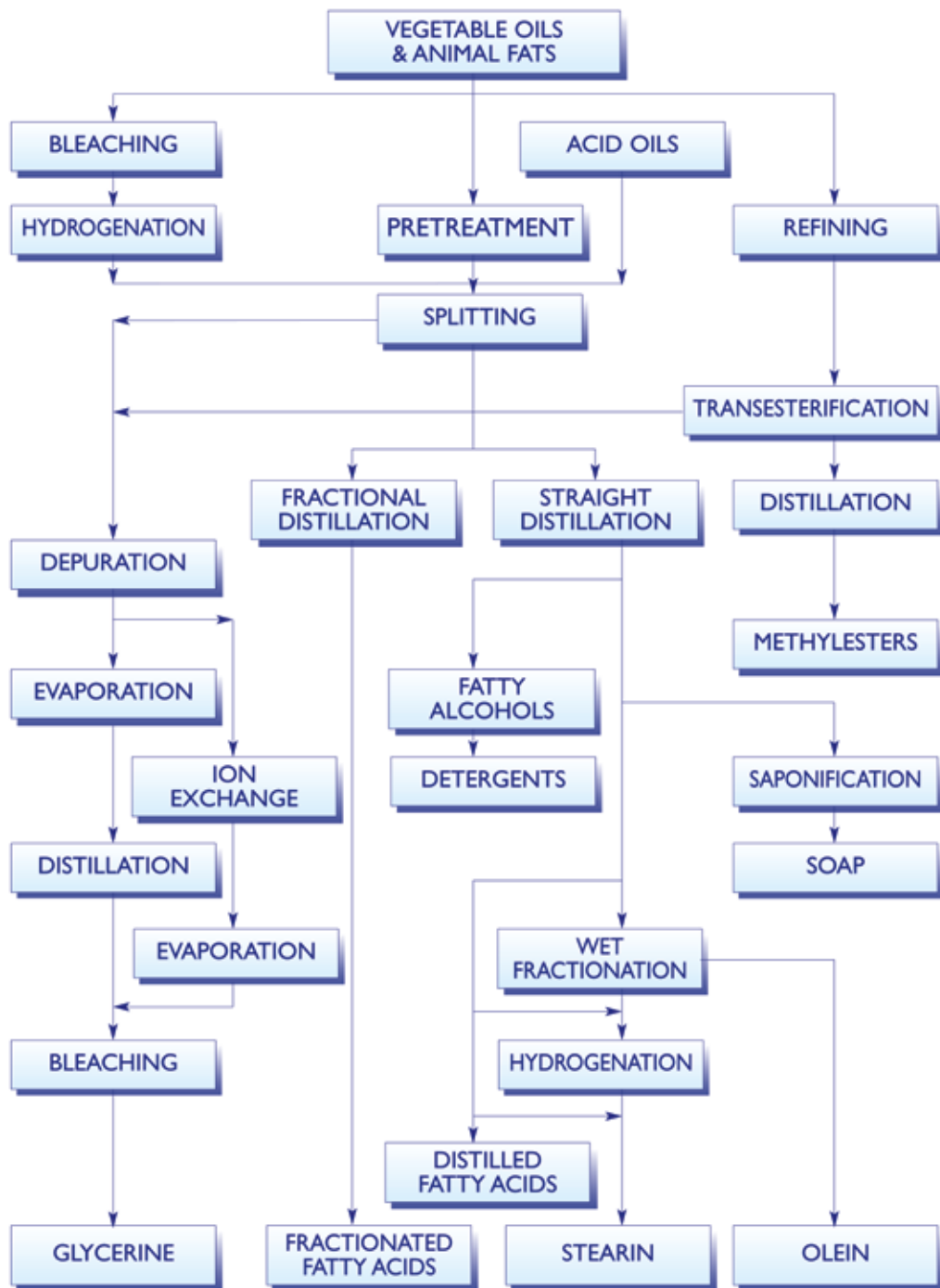
FATTY acids TECHNOLOGY



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FATTY ACIDS TECHNOLOGY



1. FATTY ACIDS TECHNOLOGY

The natural oils and fats, both of vegetable and animal origin, are worldwide used as source of fatty acids, glycerine and their derivatives.

The importance of fatty acids in chemical industry is well known and continuously increasing. They are used in a very wide range of industrial applications with thousands of end-products mainly in the following fields:

- SURFACTANTS
- SOAP & DETERGENTS
- EMULSIFIERS
- LUBRICANTS
- COATING & PAINTS
- COSMETICS
- PLASTICIZERS
- TEXTILE CHEMICALS
- RUBBER CHEMICALS

Whatever the final derivate (ester, salt, alcohol, sulphate, sulphonate, amine, amide, etc) the intermediate steps always involve the production of FATTY ACIDS, as a distilled mixture or as pure fraction: the basic steps in fatty acids technology are shown in the previous block diagram.

GIANAZZA International has a very long and proven experience in the technology of fatty acids and can supply most of the plants mentioned in the block diagram, that are individually described in the next pages, namely:

- PRETREATMENT
- SPLITTING
- STRAIGHT DISTILLATION
- HEATING SYSTEMS
- FRACTIONAL DISTILLATION
- WET FRACTIONATION
- HYDROGENATION
- FLAKING
- GLYCERINE RECOVERY



*"UNITOWER" fats splitting and distillation plant
Capacity: 100 t/d*

2. PREtreatment

PURPOSE

The main purpose of pretreatment is to remove from crude fats & oils those impurities (organic and mechanical) that cannot yield acids during splitting.

The benefits of pretreatment will be noted, at last, as a higher yield of distilled fatty acids that is a lower loss as residues from distillation.

FEEDSTOCK

Any crude fat should be submitted to pretreatment theoretically, but, in practice, oils and fats of very good quality are directly sent to splitting.

In case of bad quality feedstock, pretreatment is recommended.

PROCESS CONDITIONS & YIELDS

For good and medium quality feedstock, pretreatment can be avoided or reduced to simple water washing or reaction with a weak acid.

Filtration with bleaching earths is sometimes carried out.

For lower quality fats, the most widely used pretreatment involves reaction with a strong acid (sulphuric acid), followed by several water washings. A proper pretreatment should be capable of removing from 80% to 90% at least of undesirable impurities.

UTILITIES & CHEMICALS REQUIREMENTS

- Sulphuric acid at 96% concentration is typically used in quantities depending on the nature of feedstock (few Kg/t).
- Sodium carbonate is used to neutralize the residual acidity.
- Process water for washing: 0,15 m³ max. per ton of fat.
- Reduced quantities of low pressure steam (for heating and stirring) and electric energy are also required.

MATERIALS OF CONSTRUCTION

All parts in contact with sulphuric acid are made of special reinforced resins.

When using centrifugal separators, they are made of special grade stainless steel.

PLANT CAPACITY

Standard plants with capacity up to 100 t/24 h are batchwise operating.

For higher capacities, semi-continuous or fully continuous plants can be proposed.

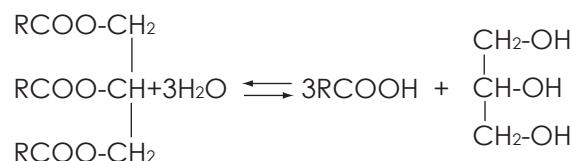


Oleochemicals plants (South Korea)

3. SPLITTING

PURPOSE

The splitting or hydrolysis reaction is carried out in order to obtain from triglycerides the corresponding crude fatty acids and crude glycerol dissolved in the excess water.



FEEDSTOCK

Any triglyceride can be submitted to splitting; in industrial practice, the most widely used feedstock are tallow, lard, palm oil, coconut oil, palmkernel oil and acid oils from refining.

When feedstock are not of first quality, it is necessary to pretreat them in order to remove organic impurities (gums, proteins, waxes, unsaponifiable matters) and mechanical impurities. The process water to be used for the reaction should be fully demineralized or, at least, softened water.

Care must be taken to avoid the presence of polyethylene in the feedstock (100 ppm max.).

PROCESS CONDITIONS

GIANAZZA International splitting plants carry out thermal splitting under pressure, with no use of any catalyst or mechanical agitation for improving the contact between the phases.

The adopted temperatures are sufficiently high (230 to 265°C) to obtain a good mutual dissolution of the water phase into the fat phase, so that the hydrolysis reaction takes place with very high conversion rate.

According to the capacity, mainly, but also depending on the quality of feedstock, GIANAZZA International can supply three different types of splitting plants:

- **BATCH TYPE SPLITTING**
- **CONTINUOUS SPLITTING, MULTICOLUMN TYPE (CONTIPERCOL)**
- **CONTINUOUS SPLITTING, SINGLE COLUMN TYPE (UNITOWER)**

BATCH TYPE SPLITTING

It is a pressure splitting plant, generally equipped with one splitting column, completed by a seal-less recycling pump. Operating pressures are in the range of 25 to 30 bar and operating temperatures are from 200 to 230°C.

Generally two separate additions of process water are carried out, in order to reach an acceptable splitting degree.

Any kind of feedstock, even in small quantities, can be processed in this type of plant; quality changeovers are not a problem.



"CONTIPERCOL" continuous fats splitting plant

3.1 SPLITTING

SPLITTING

“CONTIPERCOL” SPLITTING PLANT

The typical characteristics of this plant are summarized as follows:

- continuous, counter-current percolation system using two or more columns in series through which the fat phase is passing by overflow and the water phase is transferred by seal-less pumps.
- Typical operating pressure is 30 bar at corresponding temperature of 230°C approx. with retention time in the range of several hours.
- Heating of reactants (both fat and water) is indirect and it can easily be made by using liquid phase diathermic fluids. So there is no need of high pressure steam.
- The sensible heat of products (fatty acids and sweet waters) is entirely used to pre-evaporate the sweet waters that can be discharged with glycerol concentration of 20% or higher.
- Any type of fat can be processed.
- One or more columns, if necessary, can be used batchwise for processing small batches of particular feedstock.
- Capacity from small to medium range.

“UNITOWER” SPLITTING PLANT

This plant has the following characteristics:

- continuous, counter-current percolation system using one single column, whose upper part is used for heat recovery between the incoming water and the outgoing fatty acids, while the lower part is used for heat recovery between the incoming fat and the outgoing sweet waters.
- Typical operating pressure is 55 bar at temperature of 260°C approx. with retention time in the range of few hours.
- Higher splitting degree with reduced consumptions, but lower glycerol concentration in the sweet waters.
- Heating is carried out by means of high pressure steam being lost (that is directly injected into the splitting column).
- Equipped with advanced instrumentation for automatic control of operating conditions.
- Frequent quality changeover is less recommended due to the required time.
- Highly unsaturated fats are not recommendable as feedstock (typical feedstock are “saturated” fats such as tallow, palm oil, coconut oil).
- Typical capacities are from medium to high range.

PRODUCTS QUALITY & YIELD

BATCH SPLITTING

The splitting degree can be in the range of 95 to 97%, but it can also be lower or higher, according to the quality of feedstock and operating conditions, mainly the quantity of process water being used.

Glycerol in the sweet waters can be in the range of 5 to 15% according to the feedstock.



“UNITOWER” continuous fats splitting plant
Capacity: 120 t/d

3.2 SPLITTING

“CONTIPERCOL” & “UNITOWER”

The splitting degree is normally in the range of 98% or more and higher when processing good quality tallow, palm oil and lauric feedstock.

The sweet waters concentration is typically in the range of 15 to 20%. By using the flash vapours as heating medium in an additional evaporator under vacuum, the sweet waters can be discharged at 30÷40% glycerol content without any additional energy consumption.

As to the YIELD, the crude fatty acids yield depends on the nature of feedstock and splitting degree: for good tallow and palm oil, the yield of fatty acids is in the range of 95 to 96% of feedstock (94 to 95% for lauric feedstock), while the yield of glycerol is in the range of 10% (12 to 13% for lauric feedstock). From a theoretic point of view, the higher is the splitting degree the lower is the fatty acids yield.

SPECIFIC REQUIREMENTS

The following typical approximate consumption figures can be obtained, referred to 1 ton of crude fat (typically tallow):

	CONTIPERCOL	UNITOWER
Steam 4 & 13 bar	60 kg	50 kg
Steam 60 bar	--	160 kg
Thermal Energy (through diathermic fluid)	200.000 Kcal	--
Process Water, 70°C	0,75 m ³ (*)	0,60 m ³ (*)
Cooling Water, 25°C (without additional evaporator)	20 m ³	15 m ³
Electric Energy	8 kWh	10 kWh

(*) actual consumptions of fresh water are much lower because the recovered condensate from flash vapours are normally recycled to the process water feed tank.

MATERIALS OF CONSTRUCTION

For the splitting columns of BATCH and “CONTIPERCOL” type plants, massive stainless steel AISI 316Ti or duplex stainless steel are used.

For “UNITOWER” column, higher grade can be used, especially when processing lauric fats, such as AISI 317L or AISI 317LN (massive or clad).

Other parts of the plant working at lower temperatures are made of AISI 316L stainless steel.

STANDARD CAPACITIES

- BATCH type plants are recommended up to 30 t/d.
- “CONTIPERCOL” type is typically covering the range from 25 to 100 t/d.
- “UNITOWER” type is recommended for capacities from 100 to 300 t/d.

PROCESS DESCRIPTION

The next page shows a typical flow diagram of Contipercol and Unitower splitting plant respectively.

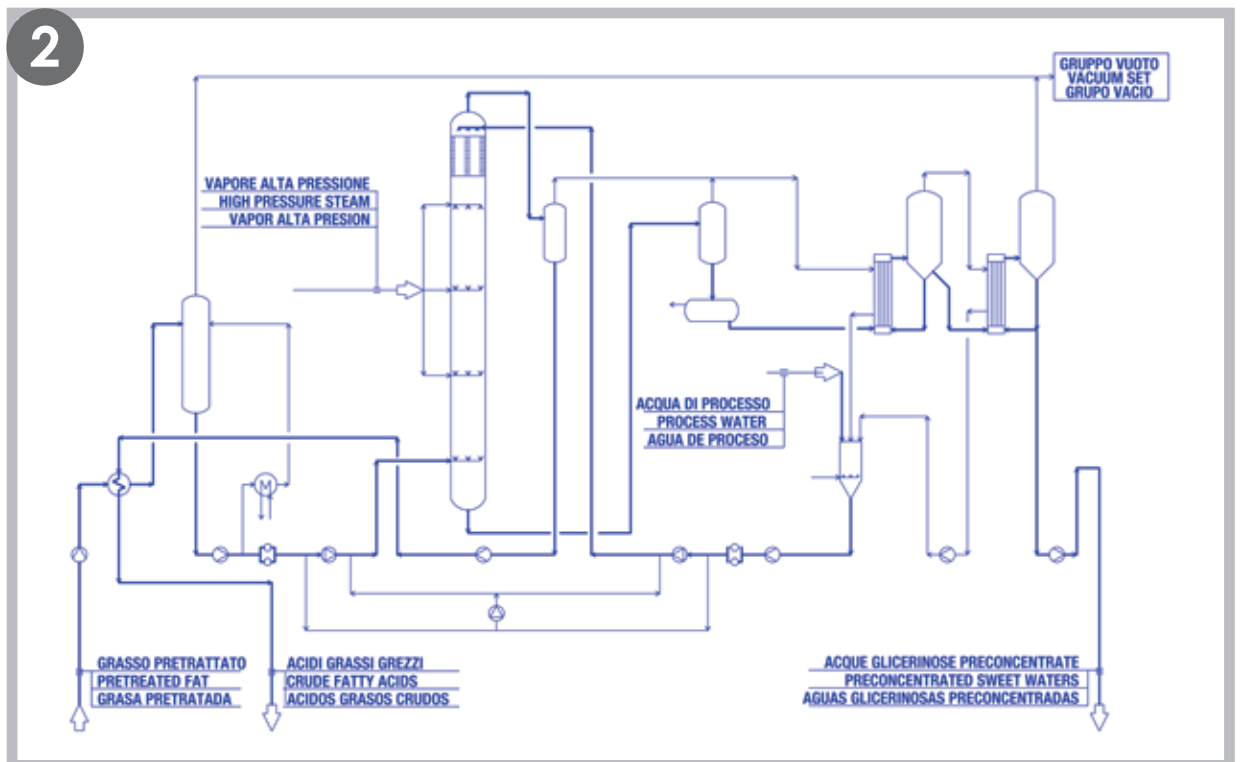
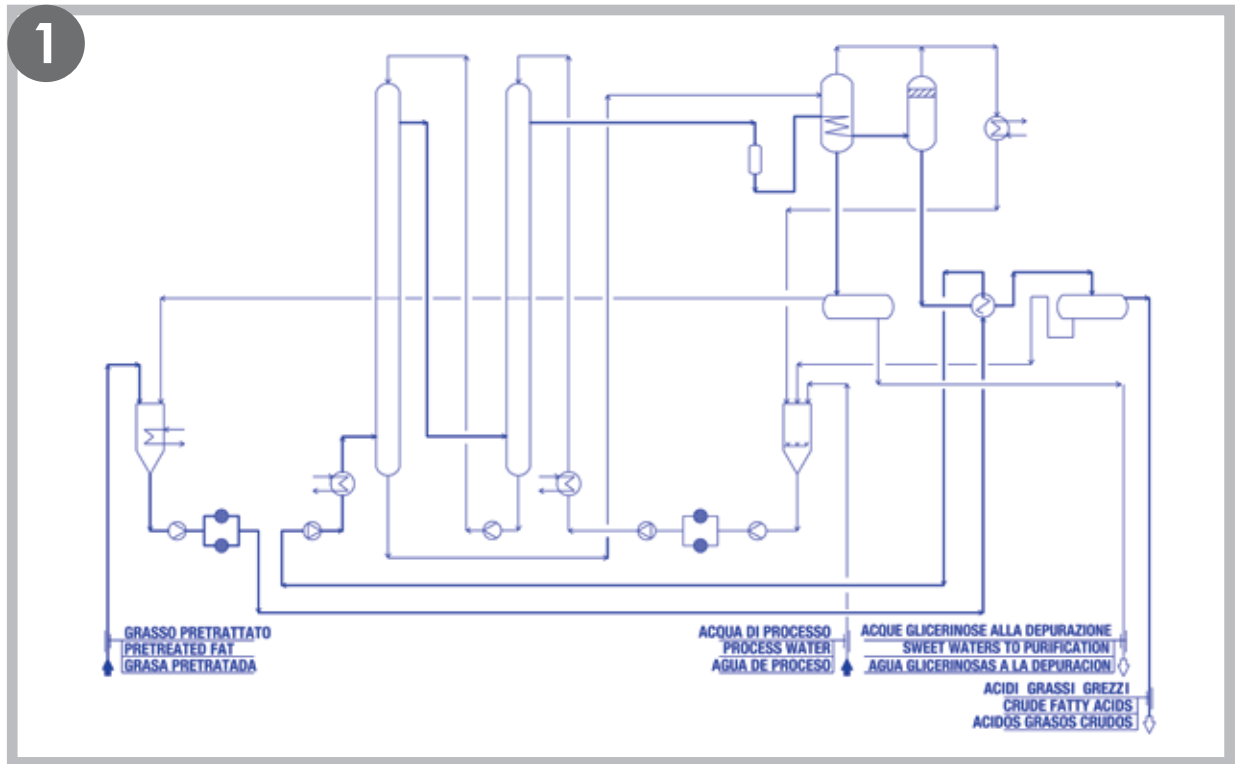
To be noted:

- while the “Contipercol” plant can be directly heated by diathermic fluid at 270°C, the “Unitower” plant needs 60 bar steam for heating, directly injected into the splitting column;
- the “Unitower” column, in its upper part, contains a set of special plates for heat recovery between the outgoing fatty acids and the incoming water.

3.3 SPLITTING

SPLITTING

In both systems the vapours obtained from the products flashing can be used to pre-concentrate the sweet waters in single or multiple effect evaporators working under vacuum.



1 - "CONTIPERCOL" continuous fats splitting plant (schematic drawing)

2 - "UNITOWER" high pressure continuous fats splitting plant (schematic drawing)

4. STRAIGHT DISTILLATION

PURPOSE

Crude fatty acids from splitting contain many impurities (mainly neutral fat and unsaponifiable matters) and are dark in colour, so that they must be purified in order to obtain a pure product with light colour and good stability.

Distillation is adopted for obtaining this result, the impurities being separated as undistilled residue (sometimes sent to further splitting in order to improve the yield of fatty acids).

FEEDSTOCK

Crude split fatty acids from different fats and oils are typical feedstock for straight distillation plants.

It is recommended that feedstock has a minimum splitting degree of 96% in order to reduce losses in the residues, but fatty acids with even lower splitting degree can be distilled, too.

The same plant can also be used for straight distillation of other fatty matters such as methylesters and fatty alcohols.

PROCESS CONDITIONS

Standard Plant.

Due to their sensitivity to heat, fatty acids must be distilled at low temperature (200 to 250°C) and, therefore, under high vacuum (5 mbar or lower). The retention time must be as short as possible; important liquid columns over the heating elements must be avoided so that vaporization is allowed only in high vacuum.

Much care, therefore, must be dedicated to the distillation column and accessories design.



GIANAZZA International column has the following features:

- reduced contact time.
- Falling film evaporators are often used as heating elements (no stripping steam required).
- Special heating candles with high heat transfer enabling to process any kind of feedstock (even of low quality) are also used.
- Small quantities of stripping steam improve distillation and protect the product against local overheating.
- Scrubbing of distilled vapours by liquid reflux strongly improving the colour of distillate.
- Hot condensation system (assisted by controlled temperature enclosed circuit), so that the distillate is leaving the condenser just few degrees below the boiling temperature and low boiling products are prevented to condense.
- High heat recovery, with production of low pressure steam.
- Vertical design of main still, therefore space saving.

4.1 STRAIGHT DISTILLATION

MATERIALS OF CONSTRUCTION

Stainless steel AISI 316L grade with good resistance is generally adopted. Sometimes, when handling short chain acids such as C6 to C12, special grades of stainless steel are used, with higher molybdenum content, for parts working at temperatures higher than 200°C.

TOPPING COLUMN

In some cases (for production of particular products) it may be requested to remove, from the fatty acids mixture, the low boiling fatty acids.

Typical cases are:

- removal of C6-C10 fraction from coconut oil fatty acids in order to produce C12-C18 cut.
- Removal of fatty acids up to C14 from tallow fatty acids in order to produce C16-C18 cut.

This problem is solved by using a fractionating column (TOPPING COLUMN) also working under vacuum, in addition to the standard main still.

STANDARD CAPACITY

Fully continuous plants are designed for capacities from 20 to 300 t/d. Higher capacities can be designed for special cases.

PRODUCTS QUALITY & YIELD

The straight distilled fatty acids will have a purity (as ratio of AV to SV) of 99% at least and unsaponifiable content of 1% max. According to the nature of feedstock, the colour will range from 0,5R to 1,5R (Lovibond, 5^{1/4}" Cell).

When distilling hydrogenated fatty acids and stearin, the final colour can be even lower, in the range of 0,1R (Lovibond, 5^{1/4}" Cell).

As to the YIELD, the distillation process has some losses (in the range of 0,1 to 0,2%, the higher figure being met when distilling lauric feedstock) and a RESIDUE that can be estimated by the following formula:

$$R (\%) = (100 - \text{FFA of feedstock}) \times 1,8 \div 2$$

The acidity of residues is normally in the range of 5 to 15% FFA max.

The LIGHT ENDS fatty acids are generally in the range of 0,5% up to 2% of feedstock (the higher figure being obtained when processing lauric feedstock).

The final yield of distilled fatty acids is therefore given by:

Feedstock - (Moisture + Losses + Light ends + Residues)

For example, when distilling tallow fatty acids with a splitting degree of 98% and 0,2% moisture, the final yield of distilled fatty acids including light ends will be in the range of 95,7% or 95,2% without light ends.



Falling film evaporator - details

4.2 STRAIGHT DISTILLATION

SPECIFIC REQUIREMENTS

Typical approximate consumption figures per ton of feedstock when processing tallow fatty acids (without topping column):

- steam 4 and 13 bar (heating, stripping and vacuum set).....100 Kg
- Thermal energy (through diathermic fluid).....160.000 Kcal (*)
- Cooling water, 25°C.....20 m³
- Electric energy.....8 kWh

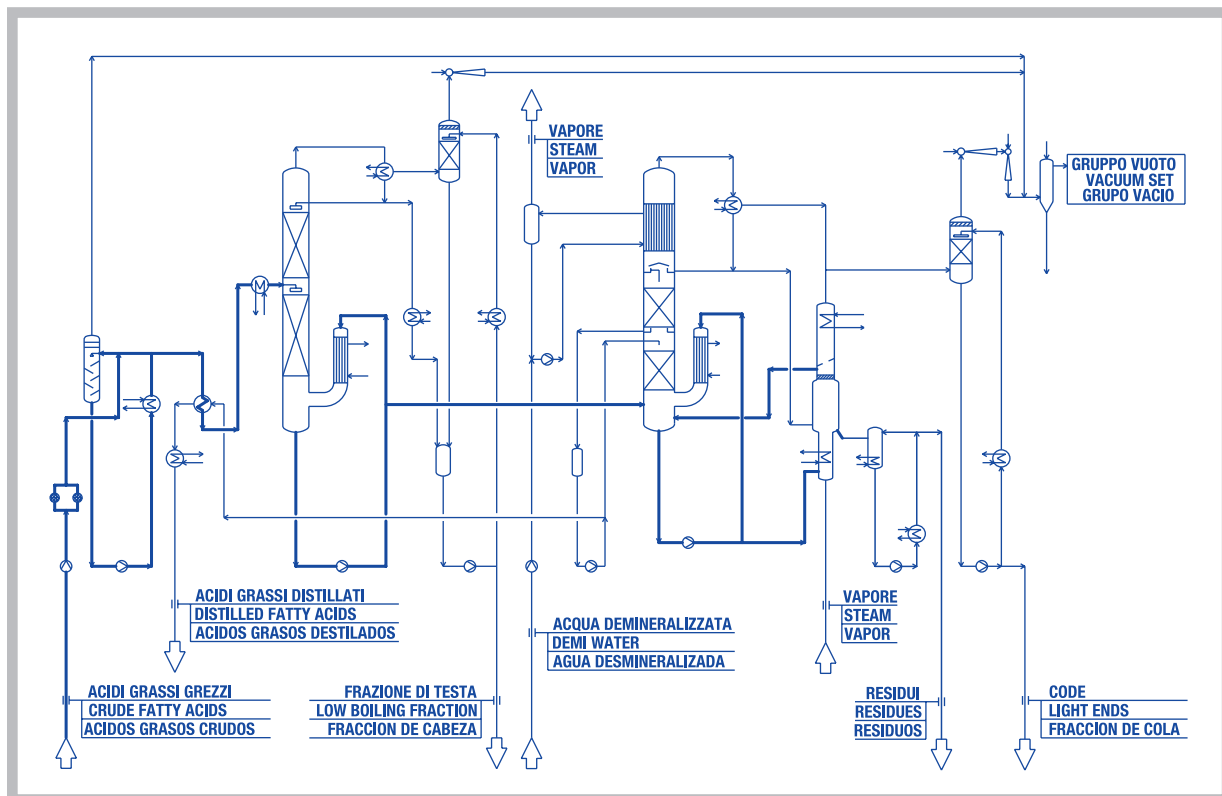
(*) to be partially recovered as low pressure steam.

PROCESS DESCRIPTION

The flow diagram shows a typical continuous plant for fatty acids distillation complete with topping column.

The following points should be remarked:

- falling film evaporators or heating candles working with live steam injection can be used as reboiler for each column, in alternative. In the attached flow diagram the falling film evaporator is used for the topping column and in the main column, while heating candles are foreseen for the residue stripper.
- Different systems of heat recovery can be adopted; the maximum entity of heat recovery is obtained by using the latent heat of fatty acids condensation for producing low pressure steam, as shown in the flow diagram.
- A separate vacuum augmenter is recommended for the topping column and the main still.
- Two or three packed beds are used in each column.
- Each column is equipped with a final condenser for the vapours leaving the column, of direct contact type, suitable to avoid any carryover of fatty matters to the vacuum set.
- All coolers in the plant are cooled by means of a tempered water enclosed circuit.



Fatty acids distillation plant with topping column (schematic drawing)

5. HEATING SYSTEMS

DIATHERMIC FLUID HEATING SYSTEM

In the fatty acids technology, both in the splitting processes and in all distillation and distillative fractionation processes, product temperatures in the range of 250°C or more have to be reached and maintained, which means that heating media at 280 to 300°C must be available. Two alternatives are possible:

- heat generators using diathermic fluid in enclosed circuit (generally in liquid phase);
- steam generators.

The obvious disadvantage of steam is the involved high pressure (80 to 90 bar), so that diathermic fluid heating systems are almost universally adopted.

The system, enabling accurate control of temperature, can be designed for several users at a time, even at different temperatures (through the use of secondary circulating pumps) and can be fully automated.

The heat generators are very compact and preassembled: the only problem is that they generally adopt burners that are not suitable for solid or heavy fuels with high viscosity.

The ideal fuel is diesel oil or domestic gas oil, with max viscosity of 1 to 2°E (at 20°C).

Methane or natural gas can also be burnt, in alternative.

COMBINED HEAT & STEAM PRODUCTION

In some cases, for example when the splitting plant is of the "UNITOWER" type, the diathermic fluid cannot be used directly as a heating medium: steam at convenient pressure is absolutely necessary because it must be injected into the process fluids.

In this case, it is still possible to avoid the use of steam boilers: the steam (with pressures up to 60 bar in special cases) can be produced in specially designed steam generators (kettle reboiler type) in which diathermic fluid is used as heating medium.

The steam generator has generally a cylindrical horizontal design, with a removable U-shaped tubular heater through which (tube side) the diathermic fluid is circulated.

PROCESS DESCRIPTION

The heating system is using an enclosed circuit with a heat transfer medium which is circulated, by means of a suitable pump, through a coil mounted inside the heat generator, which is directly heated by the flames and fumes produced by the burner.

The liquid phase diathermic fluid is pumped to the user's heat transfer equipment and it is returned to the circulating pump through a deaerator. Each user circuit can be equipped with an independent circulating pump and auxiliary temperature controller, if necessary for the process.

One of the users is a special heat exchanger which is heated by diathermic fluid and produces high pressure steam (60 bar max): this generator must be fed, of course, with demineralized water (boiler feed type) that will be transformed into steam.



Diathermic fluid heater



6. SEPARATION BY FRACTIONAL DISTILLATION

PURPOSE

The natural oils and fats are mixtures of various fatty acids, saturated and unsaturated, that must be separated into sharp fractions or even pure components in view of particular uses. Fatty acids of different number of carbon atoms have different boiling temperatures and therefore can be separated by using fractional distillation processes.

FEEDSTOCK

Crude fatty acids with splitting degree of at least 96% are the most common feedstock for this type of separation, of course.

Typical feedstock is coconut oil fatty acids, which is fractionated into C6-C10 cut, and then C12, C14, C16 and C18 fractions.

The same process and equipment can also be used for fractional distillation of methylesters and fatty alcohols.

PROCESS CONDITIONS

The operating conditions, as to temperature and pressure, are similar to those being adopted for fatty acids distillation. Live steam can also be used sometimes, according to the separation problem. Absolute pressures in the range of 15 to 40 mbar are selected for separation of low boiling fatty acids (C6 to C10), down to 4 to 10 mbar for high boiling fatty acids (C16 to C22). Typical equipment being used are:

- rectification columns of the packed type (with special structured packing), which enable very low pressure drop.
- Seal-less, canned motor or magnetic drive pumps.
- Falling-film evaporators, as reboilers, generally heated by liquid phase diathermic fluids.
- Heating candles (as used for straight distillation) when live steam is necessary.

PRODUCTS QUALITY

Very high purities and light colours can be obtained from distillative fractionation.

For example, when processing coconut oil fatty acids, individual fractions C6, C8, C10 and C12 can be obtained with purities ranging from 98 to 99% and even 99,5% in some cases.

Fatty acids with higher carbon atoms number (C14, C16 and C18) can be obtained with purities ranging from 95 to 98%. Acids C20 to C22 have generally lower purity, in the range from 90 to 95%.

The distillative fractionation is economically possible only in case of fatty acids with different carbon atoms number. At equal number of carbon atoms, the separation is not commercialized yet (for example the separation of C18:0 from C18:1) because the relative volatilities are very close to the unit, but the continuous progresses and developments in the efficiency of packing materials and accuracy of measuring instruments enable to predict that also this type of separation will be economically possible on industrial scale, in the future.



Fractionating column and falling film evaporator

6.1 SEPARATION BY FRACTIONAL DISTILLATION

SPECIFIC REQUIREMENTS

It is not possible to give even indicative figures because consumptions depend on the specific separation being considered; the requested purity affects the reflux ratio and therefore the energy requirements, so that this data must be evaluated case by case.

MATERIALS OF CONSTRUCTION

Stainless steel of different grades must be used from AISI 304 on cold parts and storage tanks up to AISI 316L, AISI 317L or higher grades steels for parts operating at high temperature with low carbon atoms fatty acids.

PLANT TYPE & CAPACITIES

There is neither standard plant nor capacity so that any specific problem is studied and engineered case by case.

In principle, three types of FRACTIONAL DISTILLATION PLANTS are available.

BATCH TYPE PLANT

Operating on small batches (not more than 20 to 25 t/d) when not more than two basic fractions are requested, besides an "odour cut" and a residue.

SINGLE COLUMN

MULTIPLE RUNS PLANT

The plant is working on fully continuous operation, but with several RUNS.

It is equipped with one fractionating column only so that, at each RUN, one fraction is obtained from the top as head product and a residue is discharged from the bottom.

The runs must be as many as the number of the wanted fractions minus one.

MULTICOLUMN, FULLY CONTINUOUS PLANT

This is the type of plant enabling to obtain the highest purity, the best qualitative results with the lowest energetic consumptions.

It consists of several columns in series; each column is separating one head fraction, the last column giving the last head fraction and a heavy fraction as bottom residue.



Fatty acids distillative fractionation plant
Capacity: 180 t/d

6.2 SEPARATION BY FRACTIONAL DISTILLATION

PROCESS DESCRIPTION

We are considering, in this case, a plant equipped with more columns (4 columns plus a stripper). In case of fatty acids coming from COCONUT oil to be processed (feedstock consisting of a mixture of C6-C18), the plant is capable of producing:

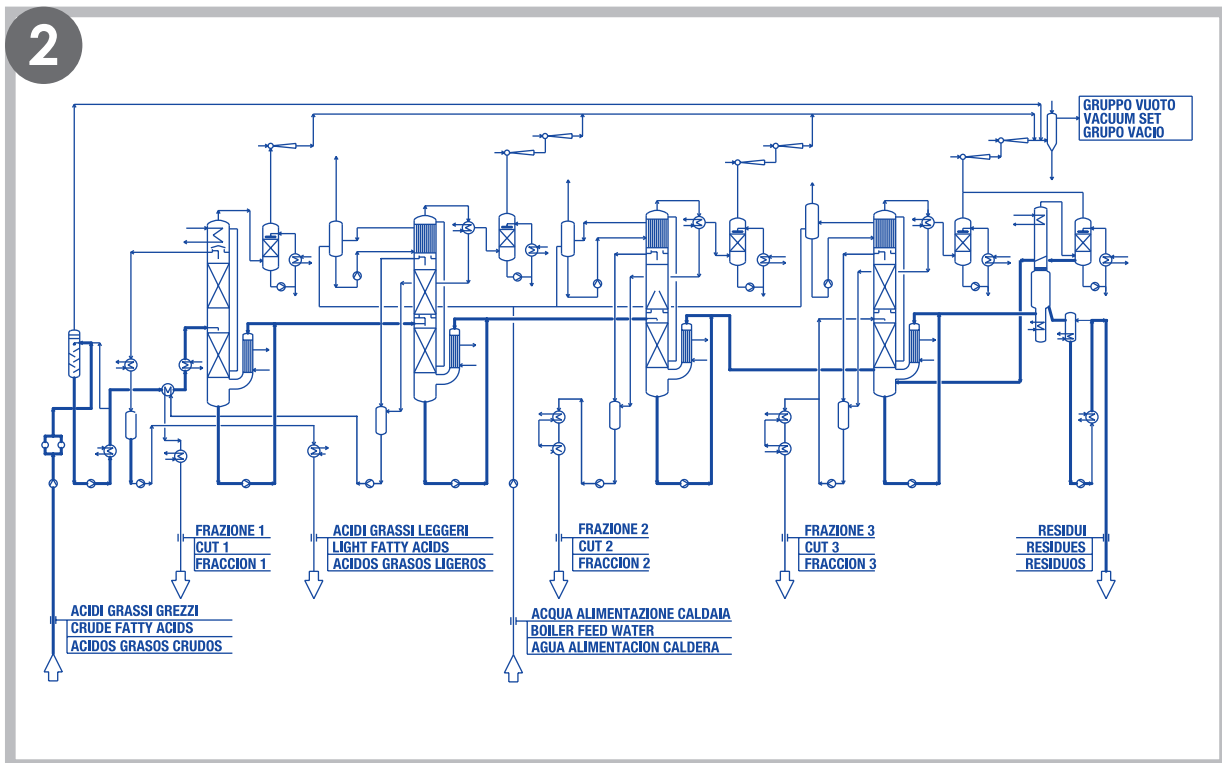
- fraction C6-C10 as light fraction from the 1st column;
- fraction C12 as head product from the 2nd column;
- fraction C14 as head product from the 3rd column;
- fraction C16 and C18 as product from the 4th column.



The stripper is designed for the distillation residue exhaustion. The columns are kept under vacuum at different levels of absolute pressure.

The columns utilize falling film evaporators as reboilers (except the stripper utilizing live steam heating elements with candles) and each one is provided with hot condenser, which produces low pressure steam by heat recovery.

- 1 - Structured packing materials
- 2 - Continuous distillative fractionation plant (schematic drawing)



7. SEPARATION BY WET FRACTIONATION

PURPOSE

It is well known that the natural fatty acids of the **saturated** type have a high melting point (that is they are normally solid at room temperature) while fatty acids of the **unsaturated** type are generally liquid at room temperature, that is they have a low melting point.

The purpose of the so called WET FRACTIONATION is to separate mixtures of fatty acids according to their melting point: in other words a liquid fraction, generally called OLEIN (predominantly but not exclusively consisting of **unsaturated** fatty acids) can be separated from a solid or hard fraction, generally called STEARIN (predominantly but not exclusively consisting of **saturated** fatty acids).

FEEDSTOCK

Although in some special cases the wet fractionation can also be carried out on crude fatty acids, GIANAZZA International recommends to use **distilled fatty acids** as feedstock for this process.

Tallow, lard, palm oil fatty acids are typically used and **generally distilled fatty acids** having:

- Iodine Value: ≥ 50
- FFA: $\geq 98\%$
- Moisture and impurities: $\leq 0,5\%$

PROCESS CONDITIONS

The fatty acids are firstly cooled down to $10\div 20^{\circ}\text{C}$ in special, scraped film coolers where the saturated fatty acids (stearin) crystallize while the unsaturated ones (olein) remain liquid. The addition of an aqueous solution of a surfactant or wetting agent enables to lower the surface tension of the crystallized acids, so that crystals are wetted by the water solution and they are dispersed into the aqueous phase, whereas the liquid acids will not be mixed with water.

It follows that the liquid phase can be separated from the aqueous phase by a centrifugal separator.

After separation, by raising the temperature of the aqueous phase, the acid crystals contained in it are melted, become liquid and immiscible again and, consequently, they can be separated in a second centrifugal separator.

The recommended wetting agent is a C10 alcohol sulphate; an electrolyte, generally MgSO_4 , is also used in the process.

PRODUCTS QUALITY & YIELDS

The composition of the two fractions obtained from the process depends on the composition of feedstock and the crystallization temperatures. The following results are typically obtained from **tallow fatty acids**:

	STEARIN	OLEIN
Yield, weight %	55	45
Iodine Value	20	78
Titer, °C	50	--
Cloud Point °C	--	4÷6

The stearin is generally sent to hydrogenation in order to reach I.V. of 1 or less. Both stearin and olein have to be re-distilled when a very light colour is requested.



Wet fractionation plant for the separation of olein/stearin

7.1 SEPARATION BY WET FRACTIONATION

SPECIFIC REQUIREMENTS

Typical approximate consumption figures per ton of tallow fatty acids submitted to wet fractionation:

- Steam, 4 bar..... 150 kg
- Process water, 80°C.....0,5 m³
- Cooling water, 25°C.....30 m³
- Electric energy.....50 kWh + refrigeration
- Wetting agent.....3 kg
- Electrolyte.....2 kg

MATERIALS OF CONSTRUCTION

All parts in contact with olein and/or stearin are made of AISI 316 stainless steel.

STANDARD CAPACITIES

Standard plants cover the range from 20 to 50 t/d. Higher capacities are designed case by case.

PROCESS DESCRIPTION

The fatty acids mixture coming from storage tanks is kept at controlled temperature at about 55°C in the first heat exchanger, from which it is pumped into the crystallizer by a pump. The crystallizer is equipped with a set of scraping blades. Cooling, taking place in a jacket, is obtained by means of direct expansion of cooling medium, coming from frigorific compressor. The partially crystallized fatty acids are then mixed with the wetting agent solution, firstly in the fast mixer and then in the slow mixer.

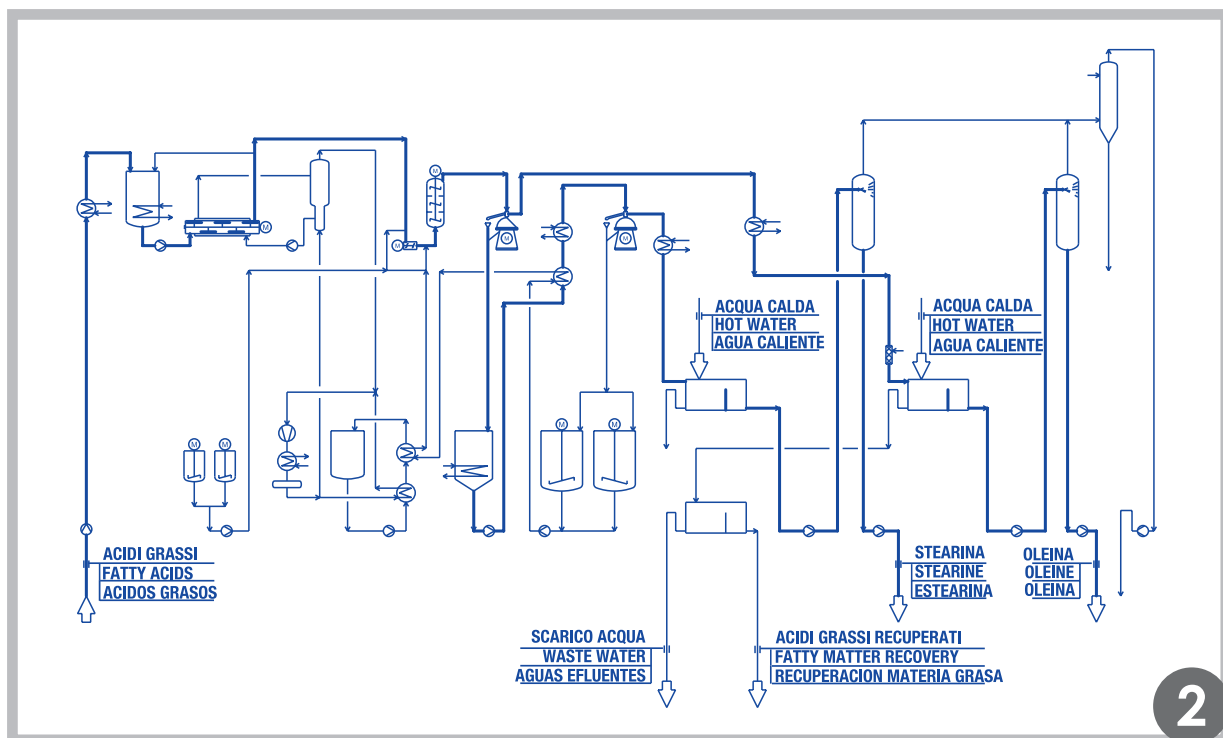
The mixture is sent to the first centrifugal separator, equipped with a special bowl, in order to avoid cloggings: the aqueous phase containing the solid fatty acids is separated from the oily phase containing the uncrystallized fatty acids (olein).

The liquid fatty acids are heated and washed with water before being dried under vacuum.

The stearin phase is also heated and sent to the second centrifugal separator: the stearin is then furtherly heated, washed with water and dried under vacuum while the wetting agent solution is cooled and recycled to the process.



1



2

1 - Wet fractionation plant - Capacity: 40 t/d

2 - Wet fractionation plant (schematic drawing)

8. HYDROgenation

PURPOSE

The hydrogenation reaction is intended to saturate the double bonds of fatty acids and decrease their Iodine Value, thus elevating their melting point, which enables to use the hydrogenated product for a wide range of industrial applications.

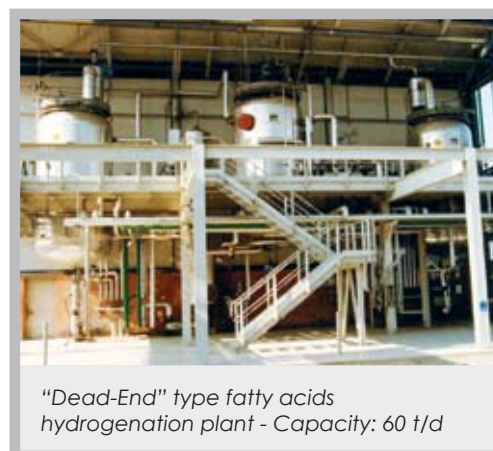
FEEDSTOCK

Typically unsaturated or partially saturated distilled fatty acids are submitted to hydrogenation. Crude split fatty acids can also be hydrogenated under special conditions.

The same plant handling fatty acids can also process pretreated oils and fats.

The reaction requires:

- hydrogen, with minimum purity of 99,8% (generally electrolytic hydrogen is used with purity of 99,9% or higher);
- nickel catalyst (the commercial products generally contain from 20 to 25% of active nickel).



PROCESS CONDITIONS

Fatty acids hydrogenation is carried out at pressures of 20÷22 bar and temperature of 200÷220°C.

Neutral oils and fats are hydrogenated at much lower pressure (3 to 10 bar).

Typical producers of fatty acids are generally processing a wide variety of products so that quality changeover and variation of operating conditions are rather frequent: very elastic plants are therefore requested and this result can only be obtained with BATCH operating plants.

When both feedstock and final product I.V. are constant, also continuous plants can be proposed.

Within the range of BATCH operating systems, three types of plants are available according to their heat recovery degree:

- **NHR (No Heat Recovery)**
The plant is equipped with one reactor only, in which the batch is dehydrated, hydrogenated, cooled and sent to filtration.
Each batch is fully processed in 6÷8 hours.
- **PHR (Partial Heat Recovery)**
The plant includes one reactor plus one receiver/heat exchanger.
This is the most widely used plant, with great elasticity.
Each batch is fully processed in 4÷6 hours.
- **IHR (Integral Heat Recovery)**
The plant includes one reactor, one receiver/drier of the batch to be hydrogenated, one drop tank for the hydrogenated product and one special heat exchanger. The plant has not only a steam consumption of ZERO but is even producing LOW PRESSURE STEAM from the heat of reaction.
Each batch is fully processed in 2÷3 hours.
This type of plant, nevertheless, is not recommended when frequent changes of feedstock are requested.

All the three types of plants are adopting a reactor that can be of two types: the "Dead-End" type or the "Loop Reactor" type.

8.1 HYDROgenation

THE GIANAZZA INTERNATIONAL "DEAD-END" REACTOR

This reactor uses a stirrer of special design (especially designed for gas-liquid reactions in presence of suspended solid catalyst), which is capable of obtaining the recirculation of gas in the liquid mass without any compressor or any other external device.

The stirrer is equipped with two types of turbines:

- the lower turbines ("flat blade" type, the number depending on the size of the reactor), give a predominantly radial circulation: this improves heat transfer, keeps the catalyst well suspended and distributes the gas uniformly; the shearing stresses of the radial liquid flow divide the gas into small bubbles and create a sort of barrier obliging the gas to stay in a prolonged contact with the fat.
- The upper turbine (double centrifugal impeller type with self-inducing hood ring) is working like a pump, which is capable of sucking the unreacted hydrogen from the top of the reactor and recycling it again into the reacting liquid mass.

"LOOP REACTOR"

In alternative to the "Dead-End" reactor previously described, GIANAZZA International can propose a "Loop Reactor" type plant for reactions at low pressure (edible oils) or for reactions at high pressure (fatty acids, fats amines, oleochemicals).

The "Loop Reactor" shown on the next page, utilizes a special pump for the recirculation of the liquid in reaction through an external heat exchanger (working as heater or cooler).

The liquid in circulation is then returned to the reactor by means of a special injector (or venturi tube) sucking the hydrogen from the upper part of the reactor and injecting and closely mixing the liquid and the catalyst through the venturi tube in the reaction product. This system is very efficient and, according to the reactor dimension, it can carry out several batches per day.

PRODUCTS QUALITY

Fatty acids and/or neutral fats can be hydrogenated totally or partially to any desired I.V. Stearin (or stearic acid) with I.V. of 1 or lower can be produced.

Advanced instruments enable, whenever requested, to predetermine the wanted quantity of hydrogen to be consumed and automatically stop the injection as soon as the prefixed amount is reached: this means to stop the reaction as soon as the wanted I.V. has been obtained. In case a very light colour is requested (0,1R to 0,5R Lovibond 5^{1/4}) a post-treatment with acid or, better, a re-distillation of the hydrogenated product should be carried out.

SPECIFIC REQUIREMENTS

Per ton of distilled fatty acids available at 70°C to be hydrogenated :

PLANT TYPE	NHR	PHR	IHR
Steam 12 bar	100 kg	50 kg	zero
Cooling water 25°C	6 m ³	3 m ³	0,5 m ³
Electric energy	8 kWh	10 kWh	12 kWh
Catalyst (at 20% nickel approx.)	1÷2 kg	1÷2 kg	1÷2 kg
Hydrogen (STP) per unit I.V.	1,1 m ³	1,1 m ³	1,0 m ³



"Dead-End" type fatty acids hydrogenation plant
Capacity: 16 t/batch

MATERIALS OF CONSTRUCTION

When processing fatty acids, all parts in contact with the product are made of AISI 316 stainless steel.

When processing neutral oils/fats, the plant can even be made of carbon steel.

As to the catalyst filter, when using filter presses of plate and frame type, the used materials are AISI 304/316 stainless steel.

8.2 HYDROgenation

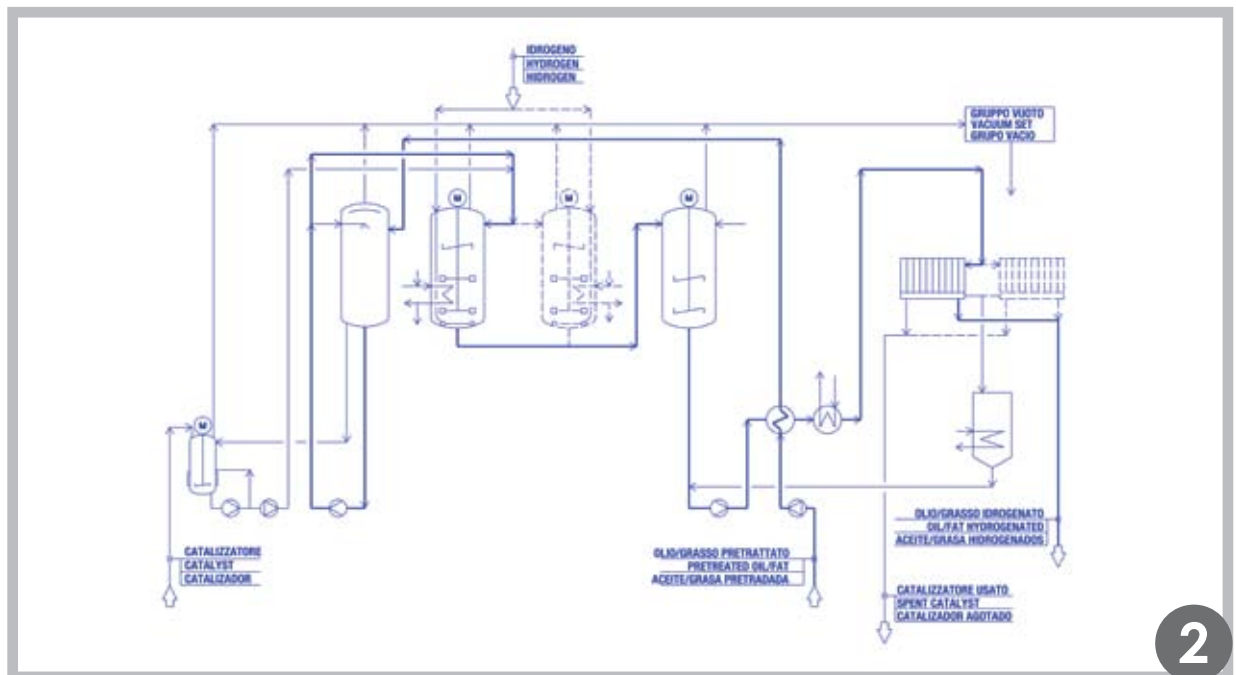
PLANT CAPACITY

Standard capacities cover the range from 20 to 250 t/d. Higher capacities can be designed case by case.

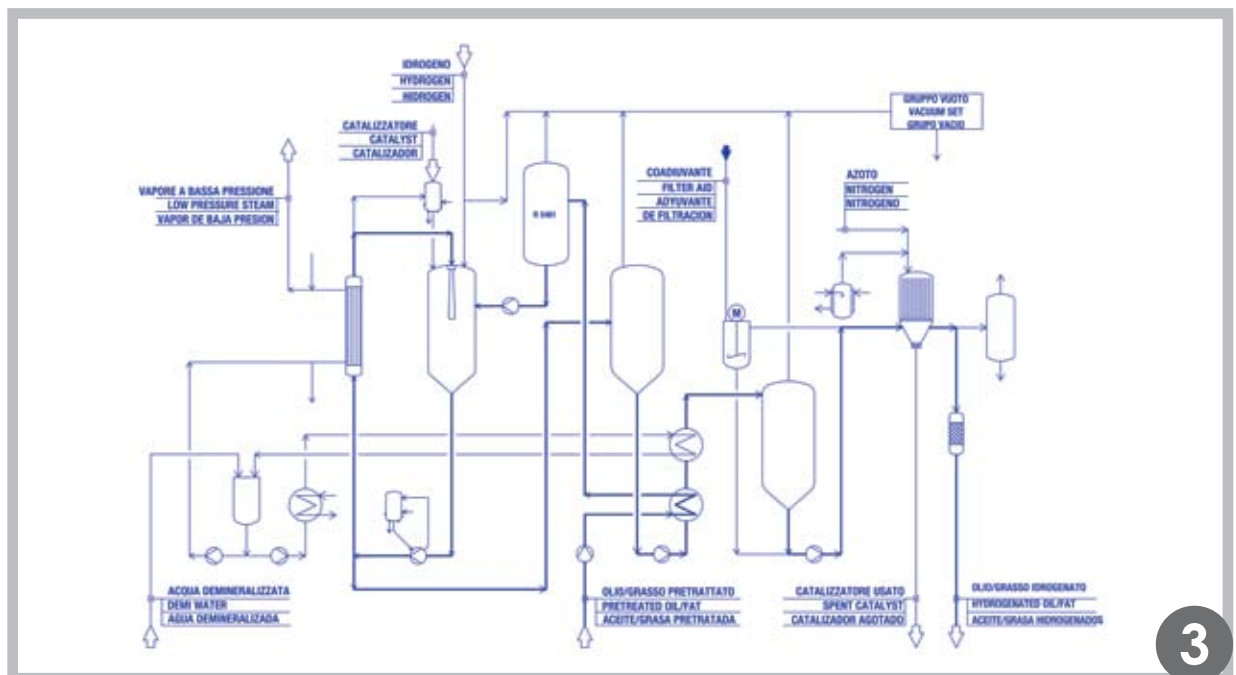
- 1 - "Loop Reactor" type fatty acids hydrogenation plant - Capacity: 250 t/d
- 2 - "Dead-End" type fatty acids hydrogenation plant (schematic drawing)
- 3 - "Loop Reactor" type fatty acids hydrogenation plant (schematic drawing)



1



2



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9. GLYCERINE RECOVERY

PURPOSE

Different processes in the oleochemical industry (splitting, saponification, transesterification) involve the obtention of by-products containing glycerine as main component.

By means of chemical and/or physical treatments, the glycerine recovery process has the purpose to produce pure glycerine to be destined to different uses (pharmaceutical, cosmetics, food etc.).

FEEDSTOCK

Three typical processes produce the three most common types of glycerol containing feedstock:

- **Sweet waters** coming from fat splitting;
- **Soaplyes** coming from soap manufacture from neutral fats;
- **Crude glycerine** coming from transesterification reaction for methylester and/or Biodiesel production.

In the three cases, the product consists of a solution containing, in different portion:

- glycerol
- water
- organic impurities (MONG)
- salts (except in case of sweet waters)

Soaplyes and crude glycerine from methylesters are therefore similar except for the latter that may also contain residual methanol.

PROCESS CONDITIONS

The basic process for glycerine recovery involves the following steps:

- Depuration
- Evaporation
- Distillation
- Bleaching

When very good quality oils and fats are processed, an alternative system can be based on ION EXCHANGE as depuration method, followed by final EVAPORATION. The typical operating conditions of each step are listed hereinafter.



Sweet waters evaporation plant - Capacity: 100 t/d

DEPURATION

Normally a chemical depuration is carried out in order to remove fatty matters and organic impurities at room pressure and temperature in the range of 80 to 90°C.

A mineral acid is used first (typically hydrochloric acid), then a flocculant is added (typically Sodium Aluminate or Ferric Chloride) and finally the mass is neutralized (typically by Sodium Hydroxide): the precipitated impurities are separated by filtration.

While, in case of sweet waters, a single final filtration is generally sufficient, a double filtration (one after the addition of the flocculant and another one after the final neutralization) is necessary in case of soaplyes.

For high capacities, fully continuous plants can be engineered.

Depuration of glycerine from methylesters demands particular processes to be studied case by case.

9.1 GLYCERINE RECOVERY

EVAPORATION

The glycerol solution has to be concentrated up to:

- 80% glycerol, in case of crude soaplyes glycerine;
- 88% in case of sweet waters;
- 80% to 85% or more, in case of glycerine from methylesters.

According to the initial glycerol content and the cost of the heating medium, different evaporation systems can be proposed, based on one, two, three or more effects, with or without thermocompression.

In case low pressure steam is available, thermocompression is not recommended.

When processing soaplyes or anyhow solutions containing salts that precipitate and have to be removed, salt boxes and salt handling equipment are used. For high capacities, centrifugal separators of special design are adopted.

DISTILLATION & BLEACHING

Crude glycerine is distilled under high vacuum to produce pure glycerine.

Distillation is carried out at temperatures of 165°C max; it is fully continuous with batchwise (alternate) stripping and removal of the residues.

The main distillation is carried out in a packed column with several sections of structured packing and special distributors.

The dimensions of the column depend on the final wanted purity and on the MONG content in the crude glycerine.

Condensation is made in two steps: the first quality distillate (95% approx of the total distillate) is obtained from a hot condensation system.

The glycerine from second condensation is sold as technical glycerine or recycled to pre-treatment.



Bleaching of the distilled glycerine consists in filtration at 80°C with 0,3% approx of activated carbon; when it is made batchwise, the filtration cake can be recycled to sweet waters pre-treatment for glycerol recovery. It can be continuous, by using granular carbon.

In case of big capacities and/or for minimum loss of glycerol, the residues stripping can be made continuously by using a special type of thin film evaporator which discharges a dry granular residue with a glycerol content in the range of 2% approx.

Glycerine distillation plant (Italy) - Capacity: 18 t/d

9.2 GLYCERINE RECOVERY

PRODUCT QUALITY & YIELDS

The distilled glycerine has a glycerol content of 99,7% min (up to 99,8%) and complies with the specifications of most international standards (EP, USP etc) and codes (Pharmacopea, Codex Alimentarius etc).

Crude glycerine of poor quality (MONG higher than 1,5%) may need sometimes to be redistilled in order to meet the a.m. quality requirements.

As to YIELDS, the distillation involves some LOSSES (expressed as % of pure glycerol present in the feedstock). The following figures can be expected:

- 2÷3% when distilling glycerine from SWEET WATERS;
- 4÷6% when distilling glycerine from SOAPLYES and METHYLESTERS (3% max when using the thin film evaporator for residues drying).

MATERIALS OF CONSTRUCTION

DEPURATION

The utilization of mineral acids causes corrosion problems. The treatment vats and related equipment are made of special plastic material (generally fiber glass reinforced polyester).

Plates and frames of filter press are made of polypropylene, pumps of acid resisting plastic or special alloys.

EVAPORATION

Supposing that feedstock has been correctly depurated and neutralized (pH from 8 to 9), the sweet waters evaporators are generally made of AISI 304 stainless steel while the soaplyes evaporators and related equipment are made of AISI 316 stainless steel or, better, of Duplex stainless steel (SAF 2205) or special alloys.

In special cases carbon steel can be used considering that the life duration of the plant will be reduced.



DISTILLATION AND BLEACHING

All the parts in contact with glycerol in the condensation system are made of AISI 304 stainless steel.

The main column, overall in case of glycerol from soaplyes, is made of AISI 316.

Structured packing is made of high grade stainless steel.

The bleaching equipment are made of AISI 304 stainless steel.

*Biodiesel glycerine distillation plant (Denmark)
Capacity: 45 t/d*

9.3 GLYCERINE RECOVERY

PROCESS DESCRIPTION

The typical flow diagram of a double effect evaporation plant for sweet waters, with thermocompressor, is shown here below.

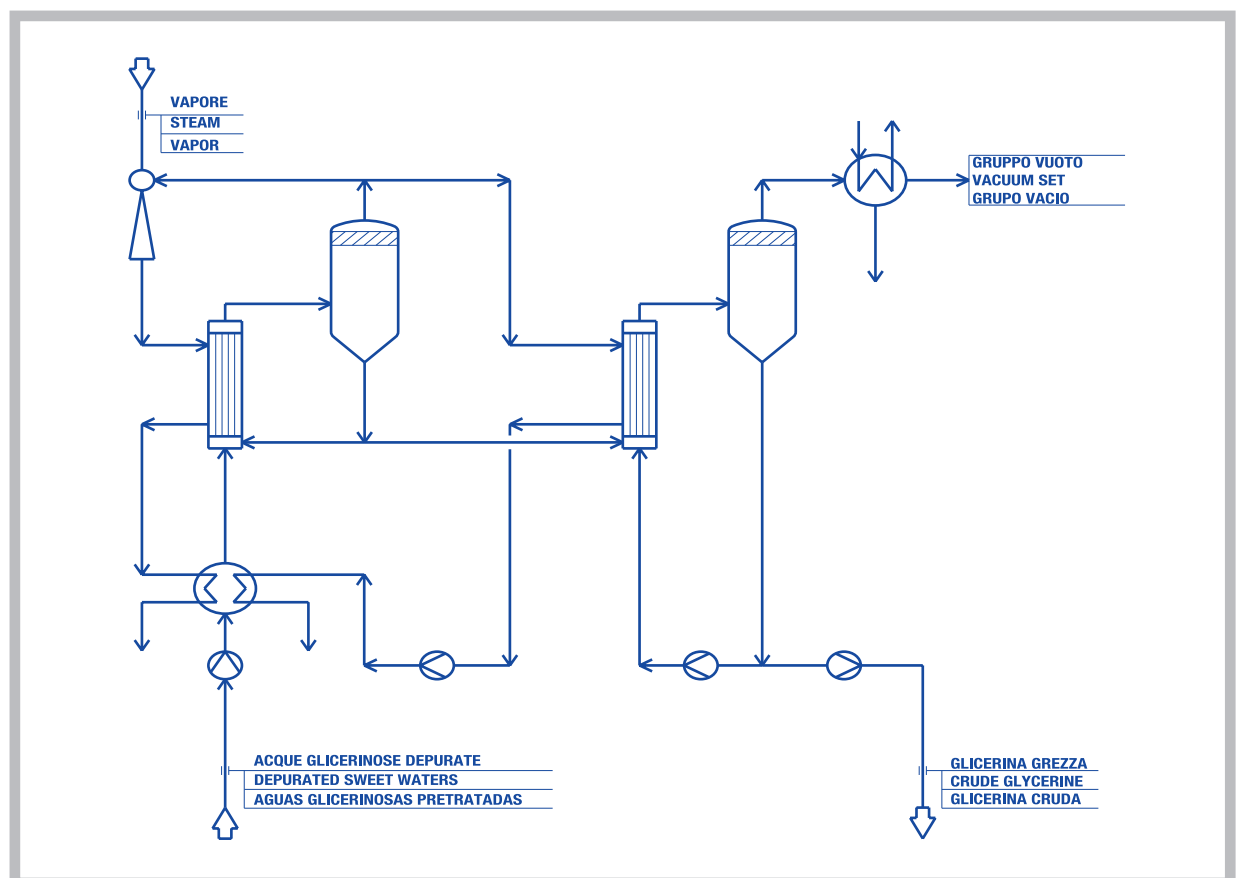
Special salt boxes (or centrifugal separators) have to be added when processing soaplyes. Crude glycerine is obtained after depuration and evaporation and, according to international standard, it has the following glycerol content:

- 88% minimum in case of crude glycerine from sweet waters;
- 80% minimum (with 10% max of salt) in case of crude glycerine from soaplyes;
- the glycerine from FAME production has glycerol content intermediate between the above mentioned figures.

The next page shows the typical flow diagram of a crude glycerine distillation and bleaching plant.

The following alternatives are possible:

- a scraped thin film evaporator can be used for continuous stripping of residues, in case of soaplyes glycerine;
- pressurized water or direct contact condensers with structured packing can be used as alternative cooling system for hot condensation;
- continuous bleaching can be supplied, based on the use of adsorption columns containing fixed beds of granular activated carbon (instead of batch bleaching by filtration).



Sweet waters double effect evaporation plant (schematic drawing)

9.4 GLYCERINE RECOVERY

SPECIFIC REQUIREMENTS

The utilities consumptions depend on the type of glycerine to be treated.

In the evaporation stage the consumptions are mainly related to the quantity of water to be removed.

In the distillation stage the consumptions are connected to the wanted final purity and the nature and quantity of MONG (the higher the purity requirements, the higher the reflux ratio being needed and the energetic consumptions).

It should be noted that the glycerine distillation plant is practically unchanged for the three types of glycerine to be processed (ex soaplyes, ex sweet waters or ex FAME/biodiesel): the differences for the three types are located at level of pre-treatment and chemical depuration while the final step (distillation & bleaching) is practically the same for all types of feedstock.

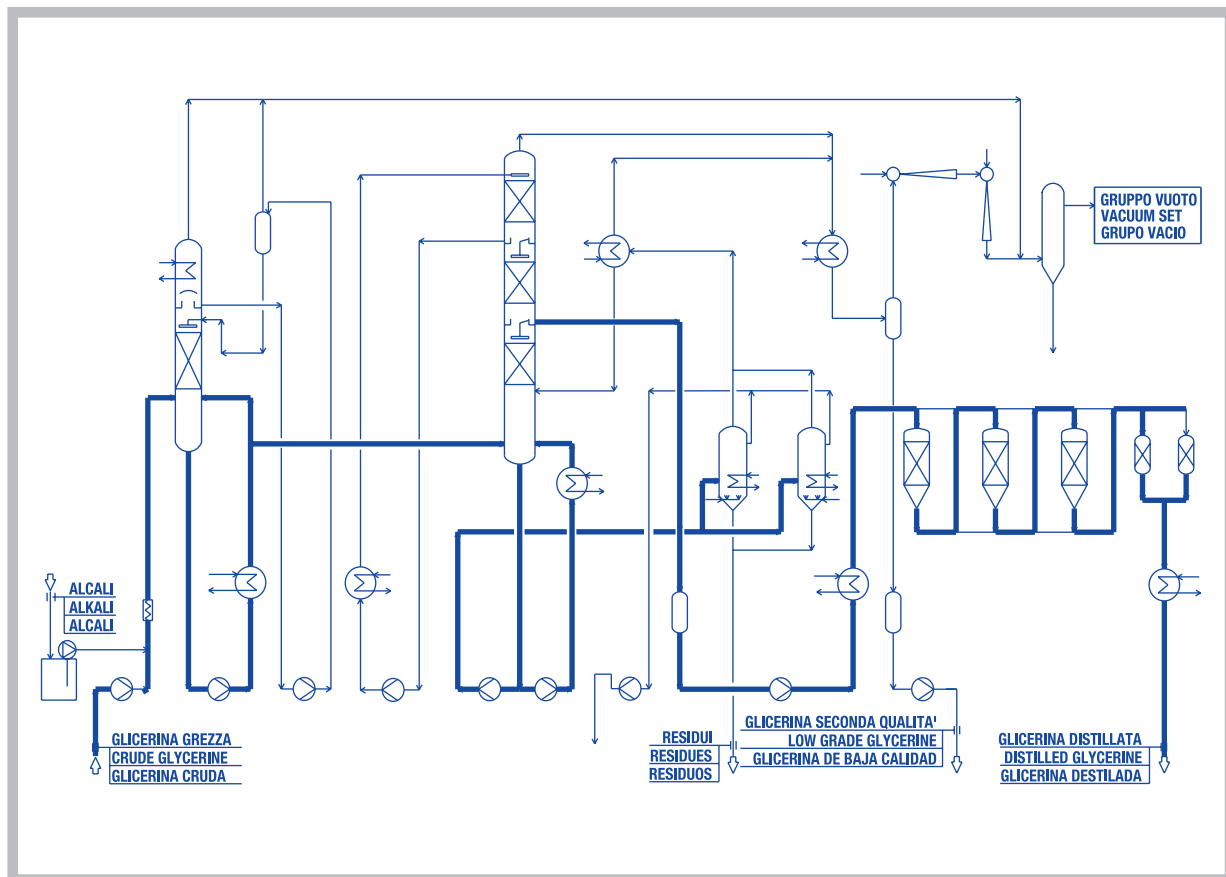
Finally, it should be noted that, in case of glycerine from FAME and according to the type of transesterification process, crude glycerine might contain peculiar salts for which the depuration process may be difficult and expensive.

PLANT CAPACITY

The depuration and evaporation plants up to 10 t/d are based on semi-continuous operation.

For higher capacities, the plants are fully continuous operating.

For glycerine distillation and bleaching, the plants are fully continuous for all capacities; for capacities higher than 50 t/d, particular solutions are available, to be discussed and agreed case by case.



Glycerine distillation and bleaching plant (schematic drawing)

Gianazza International has acquired the rights of brand Gianazza. Gianazza has realized over 250 successfully operating plants for edible oils and fats refining as well as more than 300 plants for oleochemicals throughout the world.

Considering the world continuous evolution, Gianazza International is determined to continue Gianazza's century old commitment to quality, with focus on introduction of new technologies, plants and facilities as well as providing the necessary parts needed to maintain a first class production.

Gianazza International has the technology and the know-how of Gianazza under a new structure in order to offer reliable services for years to come.

The support of skillful personnels from Gianazza ensures the continuity of more than 120 years of experiences gained since 1892.

Gianazza International is devoted to provide long term satisfaction to Customers.



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