Advances in Saponification, Drying and Soap-Finishing Technology

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Introduction

DURING THE LAST TWO DECADES the ancient art of soapmaking has been transformed into a sophisticated chemical industry. Right after the Second World War vacuum spray-drying was introduced, and its immediate success paved the way for the development of new, fully continuous, and automated processes in all phases of soap manufacturing.

The purpose of this paper is to describe the commercially available processes in fatty acid neutralization, drying, and finishing with the inclusion of some developments in 1967.

Neutralization Systems

Since the first fully automated, fatty acid neutralization plants started commercial operation in 1960 and 1961, a number of such plants have been installed. These are simple and compact since the neutralization reaction is practically instantaneous. Utility requirements are minimal because of the exothermic nature of the reaction. Precise control of the finished product specifications as far as alkalinity, moisture, electrolyte content, and additives are concerned is assured by complete automation.

Presently there are two basically different processes: De Laval's Centri-Pure system via viscosity control, and Mazzoni's SC and SCC systems via pH control.

DeLaval's Centri-Pure Process

De Laval's Centri-Pure process for fatty acids is similar to the first section of the Centri-Pure process for neutral fats, which was discussed during the last short course in 1963. There are two De Laval fatty acid systems, one for industrial soaps and another for toilet soaps. Both systems have four basic elements; heating and proportioning equipment for the raw materials, a special mixer or neutralization column, and a method to determine and control the degree of neutralization. The feedstock for the industrial soap system may range from a mixture of 85% tallow and 15% coconut fatty acids to 100% of either of these components to an assorted mixture of fatty acids from vegetable oil fats.

Fig. 1 shows the Centri-Pure process. The raw materials are heated and metered into the neutralization column by a proportioning pump. The fatty acids are injected about midway up the column into an alkaline zone, where soap has already formed and exerts an autocatalytic effect on the neutralization of the incoming fatty acids. The neutralization column is a special, patented, mixing device with an internal rotor. The neutralized mass moves toward the top of the column. Mixing within the column is aided through the use of a recirculation pump, installed in a loop which extends over the lower two-thirds of the neutralizer. The recycle rate is equal to 35 times the rated throughput of the system.

The degree of neutralization is controlled by measuring the pressure drop across the recycle pump, which reflects minute changes in viscosity. The pressure readings are fed into a differential pressure-recorder controller, which in turn causes the caustic proportioning pump to make minute adjustments as required. A constant pressure valve at the outlet of the column maintains about 50 psi of working pressure to prevent external influence on the pressure reading across the recycle pump. Various additives, as required by the producer's specifications, can be added toward the top of the column, shortly before the material leaves the unit.

The finished product is completely neutralized; however it is common practice to introduce it into an agitated, steam-jacketed pressure vessel with a 20- to 30-min retention time. Complete mixing of the additives and a homogeneous mass are ensured, together with the added flexibility of preventing scrap during start-ups and shut-downs. The product leaving the secondary tank under pressure and at approximately 275F is released to the atmosphere by spraying over chill rolls. Some drying by evaporation is accomplished before the material is fed to a hot-air band dryer. The material fed to the chill rolls will contain 77% TFA and will normally be 80–82% anhydrous, depending on the amount of water contained in the additives which are injected into the top of the neutralizer.

The toilet soap system is similar to the industrial. The differences are the design of the neutralization column and the absence of chill rolls. The liquid soap produced is moved directly into any drying system. The column is of a disc and donut design; there is a recirculation loop plus an external high speed agitator fitted into a secondary loop. It produces a less viscous 63% TFA soap.

The toilet soap column is made of 316 stainless steel, which is satisfactory for the 28% caustic used in this process. The electrolyte vs. viscosity curve for 62% TFA soap (Fig. 2) shows how, at low electrolyte content, the soap mass is extremely viscous; as the amount of electrolyte increases, the viscosity decreases to a minimum point. It rises again as the electrolyte content keeps increasing. This relationship is used to control the process.

The industrial soap column is of nickel construction to prevent corrosion from the 47–50% caustic soda. With this concentration the electrolyte vs. viscosity curve differs from the other one; no sharp minimum point can be observed (Fig. 3).

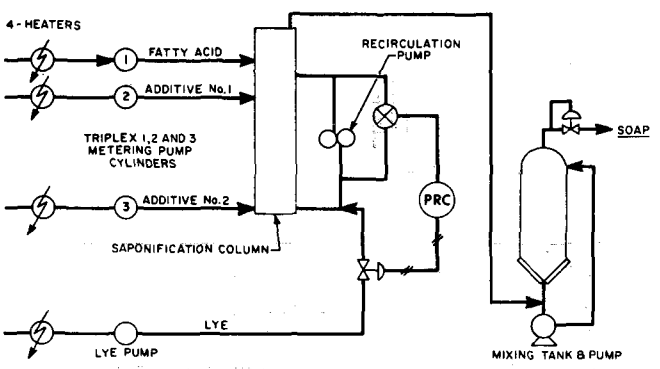


FIG. 1. De Laval centri-pure process.

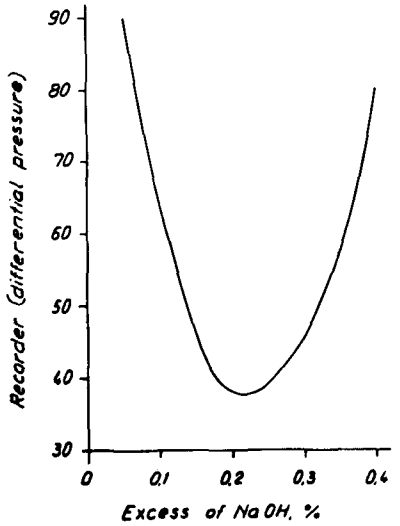


FIG. 2. Electrolyte content-viscosity curve for saponified mass of 62% fatty acid content.

Mazzoni's SC and SCC Processes

The Mazzoni Company offers two processes; one using only caustic soda as the neutralizing agent and the other using sodium carbonate and/or caustic soda.

In the SC process (Fig. 4) fatty acids and caustic soda are stored respectively in tanks 1 and 2. Both streams flow to the proportioning pump 15 through strainers 4 and 5, preheaters 9 and 10 (fitted with automatic temperature-controllers 11 and 12), and the constant level tanks 13 and 14.

For each reactant there are two pumps operating together to obtain accurate dosing. All the pumps have a common drive with a variable-speed adjustment. The stroke length of each pump is individually adjustable. The reactants enter the multistage centrifugal reactor 16 and disperse in the preformed soap coming from the first mixer 19 and the recycle pump 18.

Brine stored in tank 3 passes through strainer 6, flowmeter 7, constant level tank 8, to one of the volumetric pumps of the metering group 15. Then it is proportioned into the caustic soda circuit before entering the reactor 16. The recirculated soap passes through flow chamber 17, where an electrode assembly is mounted. In mixer 20 neutralization is completed, and small variations in alkalinity are eliminated.

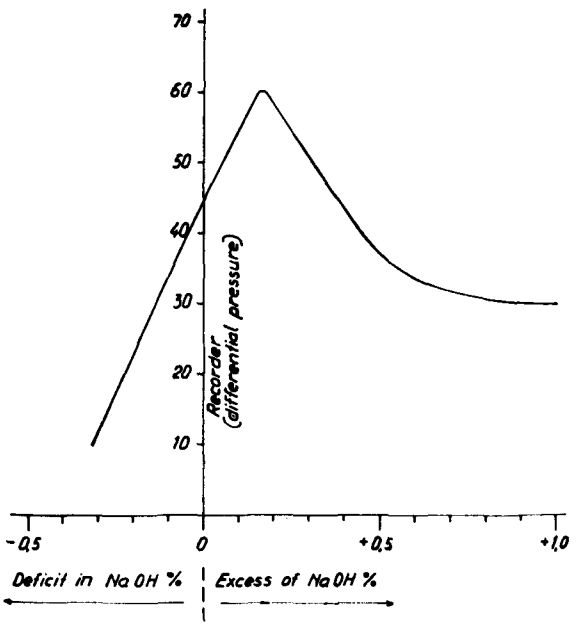


FIG. 3. Electrolyte content-viscosity curve for saponified mass of 73% fatty acid content.

In the SCC process (Fig. 5) fatty acids and soda ash flow respectively from tanks 1, 2, and 2a to the metering group 15 and on through the preheaters 9 and 10, fitted with automatic temperature controllers 11 and 12, to the contact apparatus (fatty acid neutralizer with soda ash) 13. The reaction mixture passes through a patented film reactor-separator 14, where the reaction is completed and the carbon dioxide is removed. The resulting acid soap of 68% TFA passes to the neutralizing section, which is identical to that described in the SC process. The neutralization is completed by adding caustic soda and brine from tanks 2 and 3 respectively.

Both systems are capable of processing, besides 100% fatty acid blends, admixtures of fatty acids and neutral fats in any proportion, also 100% neutral fats. The automatic control of the SC and SCC plants is based on the continuous potentiometric measurement of neat soap alkalinity. The electrode potential, measured in millivolts, is directly related to the free NaOH content of the soap. The measured potential is amplified, and the signal is used for recording and controlling the alkalinity by the automatic adjustment of the stroke length of the caustic soda pump.

Neutralization of fatty acids with caustic soda is an exothermic reaction, with about 9.5 calories of heat evolving per mole of fatty acid mixture. Soda ash neutralization, however, is endothermic with an absorption of about 4 calories per mole of fatty acid mixture. Water is also flashed off from the reaction mixture, adding the removal of carbon dioxide. The extra heat input required for neutralization is obtained by preheating the soda ash solution to about 248F and the fatty acids to 210F.

Since the soda ash neutralization reaction is reversible, a number of conditions have to be assured to drive the reaction in the proper direction. At low temperatures, carbon dioxide can react with the diluted soap solution to form sodium bicarbonate and liberate free fatty acids. The proper conditions to be met are: temperature close to 212F, low partial pressure of carbon dioxide for its easy removal, and high TFA content of the acid soap formed (about 68%).

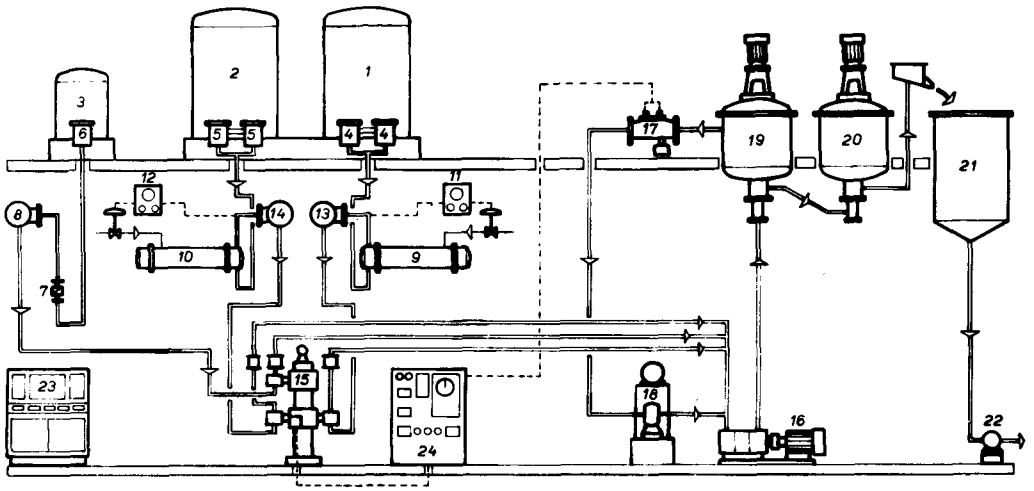
Meccaniche Moderne made available, about two years ago, a system similar to Mazzoni's SC process.

Drying Systems

Hot-air band dryers, also referred to as conveyor or flake dryers, dominated soap drying until they were challenged by vacuum spray-dryers, which made their appearance after World War II. Today there are approximately 800 vacuum spray-dryers in operation. This large number is the best proof of their rapid, world-wide acceptance.

A vacuum spray-dryer is capable of producing dry toilet-soap base, soap-synthetic base, industrial soap, filled and unfilled laundry soap in bar form by merely changing operating conditions. This versatility combined with favorable utility, manpower, and space requirements plus proper crystallization characteristics of the base or finished product account for the popularity of vacuum spray-dryers.

The following companies offer vacuum spray-dryers of similar designs: Miag, and Weber and Seelander



1 FATTY ACID FEED TANK
2 CAUSTIC SODA FEED TANK
3 BRINE FEED TANK
4 FATTY ACID STRAINERS
5 CAUSTIC SODA STRAINERS
6 BRINE STRAINERS
7 BRINE FLOWMETER
8 BRINE CONSTANT LEVEL TANK
9 FATTY ACID PRE-HEATER
10 CAUSTIC SODA PRE-HEATER
11 FATTY ACID TEMPERATURE CONTROLLER
12 CAUSTIC SODA TEMPERATURE CONTROLLER
13 FATTY ACID CONSTANT LEVEL TANK
14 CAUSTIC SODA LEVEL TANK
15 PROPORTIONING PUMPS
16 SAPONIFIER
17 FLOW CHAMBER WITH ELECTRODE ASSEMBLY
18 SOAP RECYCLE PUMP
19 FIRST STAGE MIXER
20 SECOND STAGE MIXER
21 FINISHED SOAP HOLDING TANK
22 SOAP TRANSFER PUMP
23 ELECTRIC CONTROL BOARD
24 mV CONTROL BOARD

FIG. 4. "SC" continuous fatty acid saponification with caustic soda.

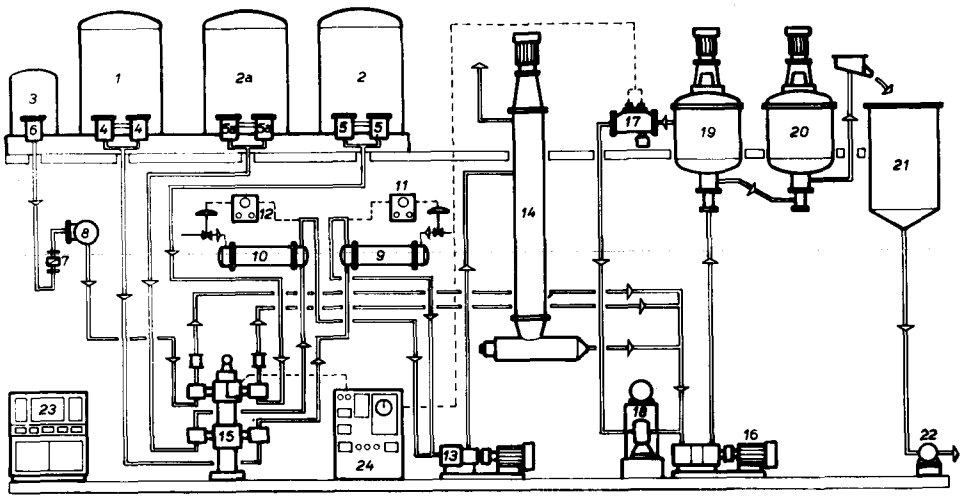
from Germany; Meccaniche Moderne, Gariboldi, and Mazzoni from Italy.

Vacuum Spray-Dryer

Mazzoni introduced vacuum spray-drying more than 20 years ago, and the dryer of today (Fig. 6) consists of a variable-speed drive pump 1, which delivers through a filter 2 into a feed tank 3 the liquid or "neat soap." From the feed tank another variable-speed pump 4 feeds a shell and tube heat-exchanger 5. The soap passing inside of the tubes is heated countercurrently by steam passing on the outside. The preheated soap enters the vacuum chamber 6 through a revolving nozzle, which sprays a thin layer of dried and partially cooled soap film on the walls of the chamber. The soap is removed by rotating scrapers and falls to the bottom of a plodder 13, which extrudes the product in the form of noodles or pellets.

Either single or double plodding is used, depending on the variety of products dried and the subsequent finishing-line layout. Water is flashed off in the vacuum chamber 6 in an amount determined by the quantity of heat imparted to the neat soap in the heat exchanger and the degree of vacuum in the system.

Vapors and soap fines pass through two powder recuperators 7 and 8. The powdered soap is separated and recovered at the bottom of the recuperators, where a powder recovery plodder 9 extrudes the fines continuously. The talcum powder-like fines can either be extruded outside of the dryer or returned



1 FATTY ACID FEED TANK
2 CAUSTIC SODA FEED TANK
2a SODA ASH SOLUTION FEED TANK
3 BRINE FEED TANK
4 FATTY ACID STRAINERS
5 CAUSTIC SODA STRAINERS
5a SODA ASH STRAINERS
6 BRINE STRAINERS
7 BRINE FLOWMETER
8 BRINE CONSTANT LEVEL TANK
9 FATTY ACID PRE-HEATER
10 SODA ASH SOLUTION PRE-HEATER
11 FATTY ACID TEMPERATURE CONTROLLER
12 SODA ASH TEMPERATURE CONTROLLER
13 NEUTRALIZER
14 CARBON DIOXIDE SEPARATOR
15 PROPORTIONING PUMPS
16 SAPONIFIER
17 FLOW CHAMBER
18 SOAP RECYCLE PUMP
19 FIRST STAGE MIXER
20 SECOND STAGE MIXER
21 FINISHED SOAP HOLDING TANK
22 SOAP TRANSFER PUMP
23 ELECTRIC CONTROL BOARD
24 mV CONTROL BOARD

FIG. 5. "SCC" continuous fatty acid saponification with soda ash and caustic soda.

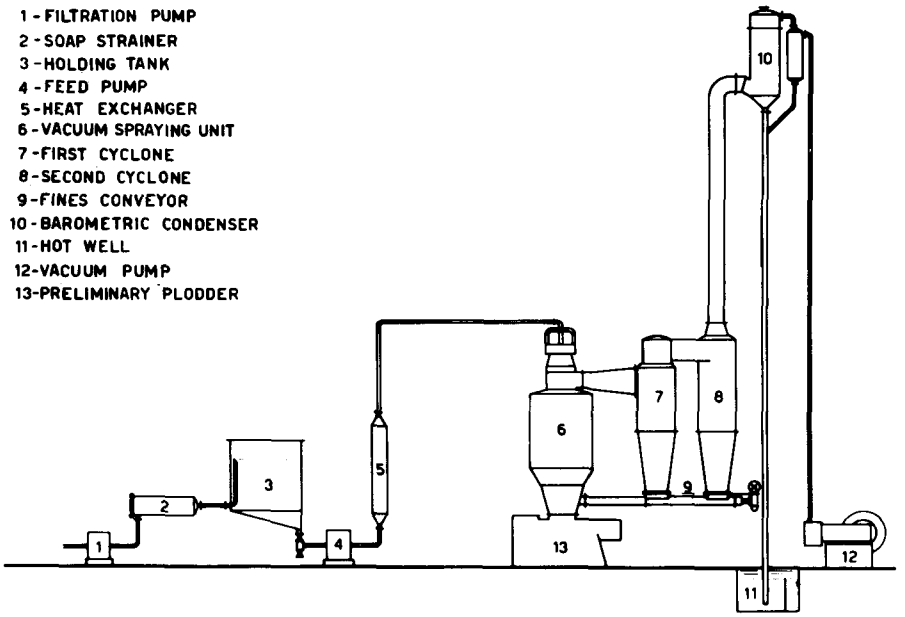


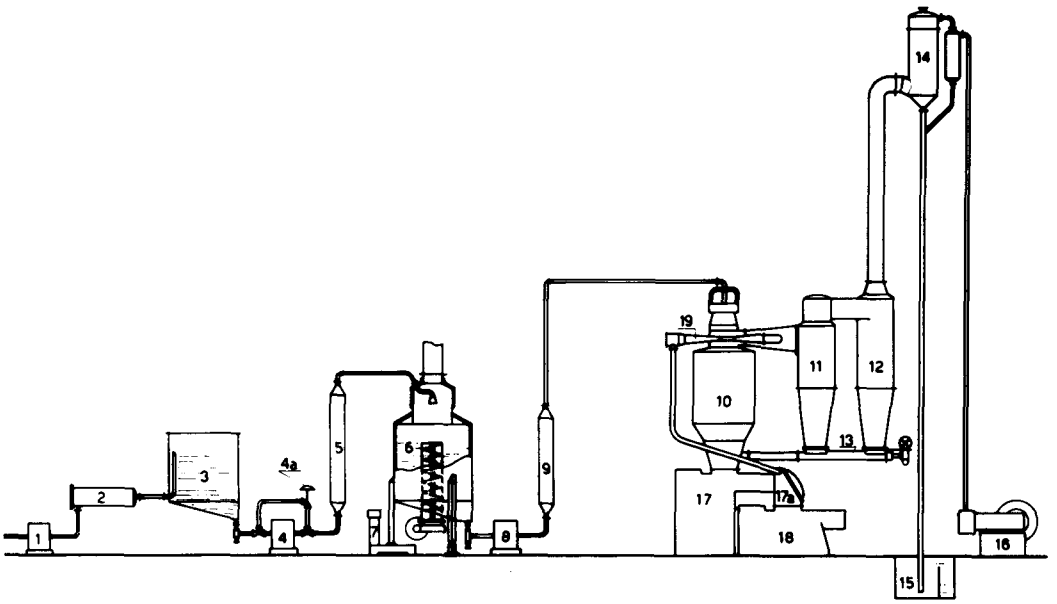
FIG. 6. Mazzoni's vacuum spray dryer.

continuously to the bottom section of the spray chamber. The vapors reach the barometric condenser 10 to be condensed and discharged into a hot-well.

Multistage Dryer

Mazzoni's latest drying system is the multistage dryer (Fig. 7). It is an expanded version of the single-stage vacuum spray-dryer just described. The new dryer is capable of processing a wider range of materials, and it also attains a higher degree of drying. Industrial soaps with 6% and lower final moisture, soap-synthetic mixtures with as high as 40% initial moisture, regular toilet soaps as well as special soaps like 100% coconut bars can be processed with the three-stage system.

First-Stage Drying. Neat soap is pumped by pump 1 through filter 2 into feed tank 3. Pump 4 sends the soap to the first shell and tube heat-exchanger 5. The preheated soap enters the atmospheric flash chamber 6 through a sepecial pressure nozzle. This chamber is provided with a mixing worm, which helps to liberate the vapors and to homogenize the partially dried soap, and pumps to the second stage. A constant level of soap is maintained in the flash chamber, which rests partially on a scale 7 and partly on the floor. The scale's pneumatic control device



1- FILTRATION PUMP
2- SOAP STRAINER
3- HOLDING TANK
4- FIRST STAGE FEED PUMP
4a- MODULATING VALVE
5- FIRST STAGE HEAT EXCHANGER
6- FLASH CHAMBER
7- PNEUMATIC LEVEL CONTROL
8- SECOND STAGE FEED PUMP
9- SECOND STAGE HEAT EXCHANGER
10- VACUUM SPRAYING UNIT
11- FIRST CYCLONE
12- SECOND CYCLONE
13- FINES CONVEYOR
14- BAROMETRIC CONDENSER
15- HOT WELL
16- VACUUM PUMP
17- PRELIMINARY PLODDER
17a- CONNECTING VACUUM CHAMBER
18- FINAL PLODDER
19- THIRD STAGE BOOSTER

FIG. 7. Mazzoni's three-stage dryer.

acts upon the control valve 4a, which lets any excess soap be recycled through pump 4.

Second-Stage Drying. Another variable-speed pump 8 via a second shell and tube heat-exchanger 9 feeds the partially dried soap into the vacuum spray chamber 10. This portion of the plant and its operation is similar to the single-stage dryer.

Third-Stage Drying and Cooling. The soap extruded by the preliminary plodder 17 in the form of pellets falls into the vacuum chamber 17a which interconnects the two plodders 17 and 18. The absolute pressure in this chamber is considerably lower than the absolute pressure in the main vacuum chamber 10. In the interconnecting vacuum chamber 17a the pellets undergo some minor additional drying and considerable cooling. The fully homogenized and compressed pellets leave the final plodder 18 and are ready for the immediate subsequent finishing operation. A small booster 19 is used to obtain the lower absolute pressure in the interconnecting vacuum chamber 17a.

Double-Expansion Dryer

In the Miag dryer (Fig. 8) drying of liquid soap containing 63% TFA to toilet soap base with 78–80% TFA or higher is achieved by a double-expansion system. Neat soap at about 190F is taken by a variable-speed pump and passed through two shell and tube heat-exchangers. Steam at about 115 psig, passing outside the tubes, heats the soap passing inside the tubes to 275F.

The soap-steam mixture expands through a nozzle into the first expansion chamber, which is kept at 0.5 at m. pressure. Water is flashed off; the semi-concentrated soap, now containing 72% TFA, cools down to 230F. In the lower section of the expansion chamber a mixer stirs the soap continuously in order to compensate for any differences in the fatty acid content.

A second variable-speed pump delivers the 72% TFA soap to another set of tubular heat-exchangers. The soap is heated again from 230F to 285F. Another expansion takes place in the second expansion chamber, which, like the first one, is equipped with a mixer in its lower section. The soap now dried to 78–80% TFA has a temperature of 230F. It is discharged onto chill rolls, where its temperature is reduced to 140F. The soap solidifies on the two counter-rotating rolls, and it is removed in flakes. Subsequently the flakes are usually plodded into pellets to increase their bulk density, facilitating storage and further processing.

Miag also offers a vacuum spray-dryer similar to Mazzoni's single-stage dryer.

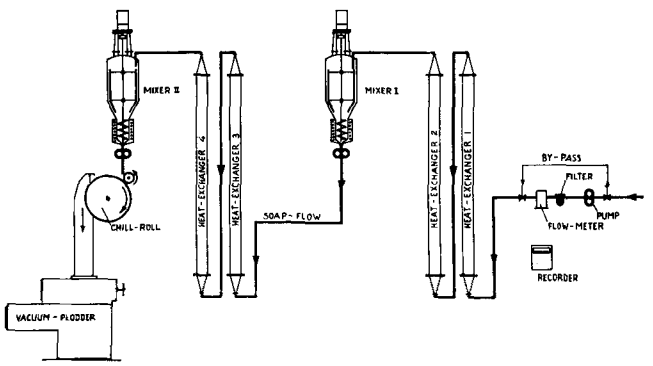


FIG. 8. MIAG-double expansion dryer.

Finishing Lines

Finishing lines for low unit-price items have to be continuous and fully automated, probably more so than many other types of finishing lines. The last decade has seen the development of fully automated, high-speed lines for different soap and synthetic products in bar form.

Automated Toilet Soap Line

Fig. 9 illustrates Mazzoni's continuous automated finishing-line type LTC. A fully automated batch doser-mixer receives all the liquid and powdered ingredients from the various holding tanks and bins. This dosing-mixing system is especially suitable for germicidal slurries which are difficult to dosify on a continuous basis with accuracy. After a predetermined mixing period the doser-mixer's bottom-opening, sliding door discharges the mixed product consisting of soap pellets and all the ingredients which the final bar will contain.

The mix is conveyed on a belt conveyor into a duplex refining plodder. Each stage of the refiner is fitted with fine screens (up to 50 mesh) and worms designed to give maximum refining action. The pellets, after undergoing two intense refining actions in the duplex refiner, proceed on another belt conveyor to a duplex vacuum plodder for final refining through another screen and air-free extrusion in a continuous slug form.

The cutting operation, which follows, has also reached complete automation with the invention of an adjustable cutter. To adjust the cutting length within certain ranges, it is sufficient merely to turn a hand wheel on the TV cutter instead of having to change the chains and thus stop the operation. The cut slugs enter a vertical conditioning tunnel to be prepared for proper stamping. These conditioners increase the production rate of high-speed finishing lines by minimizing downtime because of cleaning of the soap-press dies and also improve the over-all appearance of the stamped finished product.

The conditioned slugs then reach the stamping operation. In the soap industry there are two basically different presses: horizontol-action types, mainly for rectangular shape bars, and verticle-action types,

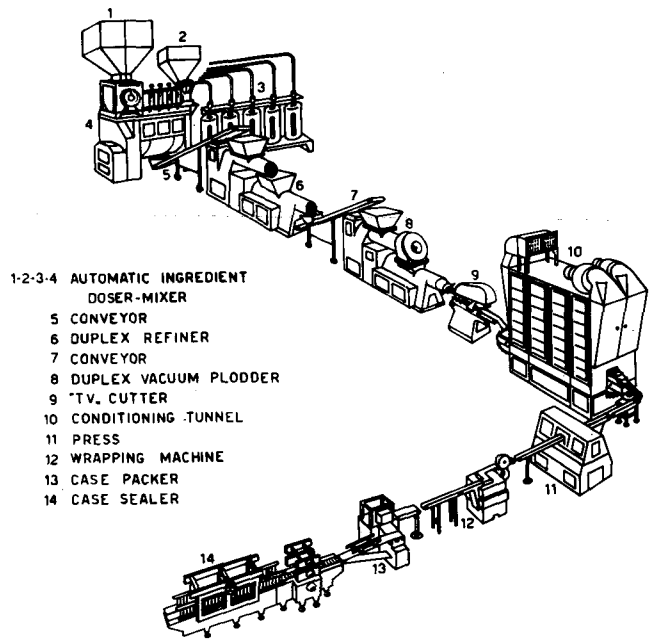


FIG. 9. Mazzoni's toilet soap processing line "LTC".

mainly for oval shapes without side bands. More than a year ago, for the first time, a new combination press was introduced. The STU press is capable of stamping out any shape. To switch from the rectangular to the oval or pillow shape bars, only the dies and the dies box have to be changed. The STU press comes in three models for stamping more than 100, more than 200, or more than 300 bars per minute. The stamped bars are moved on to the wrapping, case-packing, and case-sealing equipment. The discussion of packaging equipment is beyond the scope of this paper.

The same companies which offer vacuum spray-dryers also manufacture complete or partial finishing-line equipment.

Synthetic Laundry Bar Processing Line

The need for a synthetic detergent in bar form has been urgent since the spectacular growth of powdered detergents. A synthetic laundry bar would be a substitute for the traditional laundry or household soap, still used in large quantities throughout the world. Latest statistics show that, in Europe, laundry soap still accounts for 40% of the total soap market.

The development of a synthetic laundry bar took a considerable amount of time and effort owing to formulation difficulties and the lack of adequate processing equipment. The different characteristics exhibited by this type of product necessitated new machinery, and eventually an entirely new processing line was evolved. Synthetic laundry bars are soft, tacky, dense, and hygroscopic. They lack the lubricating properties of soap and become extremely hard rather quickly. The SLB line was formally introduced only in 1967 although the first SLB product appeared in mid-1963.

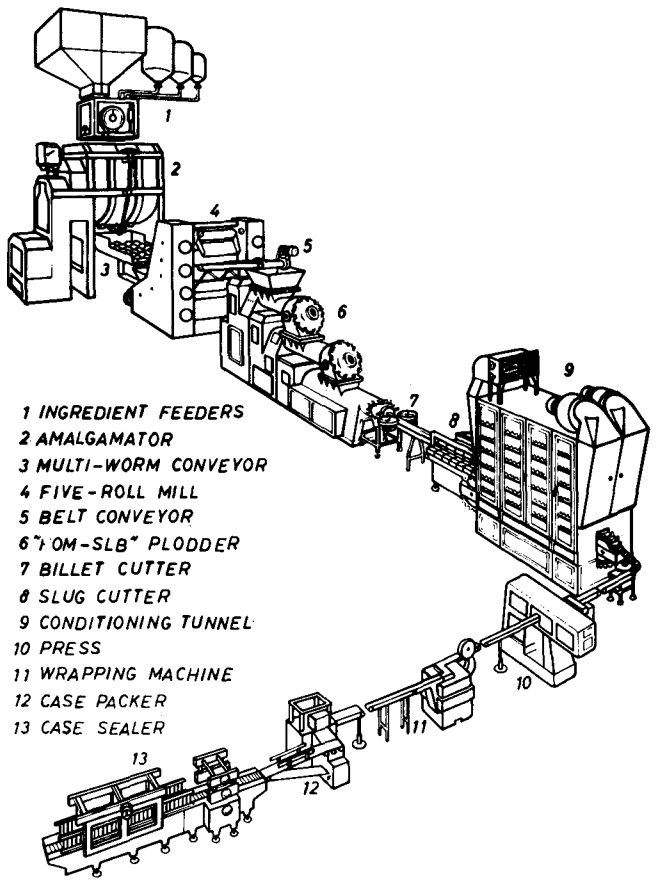


FIG. 10. Mazzoni's synthetic laundry bar processing line "SLB."

The most accepted method of producing synthetic laundry bars is by the neutralization of sulfonic acid. Powdered formulations have also been used, but, because of difficulties in controlling the homogeneity of the mixed ingredients in the rather short time allowed for the mixing cycle, their use is decreasing.

The various powdered and liquid ingredients which make up the finished product are stored in a series of silos and tanks 1 (Fig. 10). They are fed in a carefully predetermined sequence into a jacketed mixer-reactor 2 of a special design capable of handling viscous, pasty mixtures. The neutralized product is conveyed by multiworm conveyors 3 to a five-roll mill 4 for homogenizing and cooling. From the mill the material is sent to a special plodder 6, which will be described later.

The finished product extruded from the plodder is cut continuously by a rotary wire cutter 7 into billets of a certain length, which are multiple of the final bar size. The slug cutter 8 that follows is an automatic, adjustable multiple wire-cutter, which cuts the long billets into the final individual size slugs. The cut slugs or blanks proceed directly into a verticle conditioning tunnel 9, where they have to be cooled to optimum consistency for trouble-free stamping. According to prevailing temperature, humidity, and product consistency, the tunnel can be provided with an air-conditioning system. The conditioned slugs then are moved to conventional stamping and packaging equipment 10, 11, 12, and 13.

The heart of the SLB line is the FOM/SLB plodder, the latest and most versatile machine of the soap industry. The unit is a triplex plodder, consisting of three groups of twin-worm plodders connected in tandem. The first stage is a refining and feeding type of plodder. The second and third stages are special plodders which are designed to incorporate air into the product. Aeration is achieved through the combined action of a series of refining screens, pressure plates, mixing rotors, and special plodder worms. The FOM/SLB is capable of processing the following products: a) dense and light synthetic laundry bars, b) regular and floating synthetic toilet bars, c) regular and floating soap-synthetic bars, and d) regular and floating toilet soap bars.

Synthetic and Soap-Synthetic Bar Finishing Lines

Synthetic toilet bars and soap-synthetic "combo" bars are processed through finishing lines which only differ in the refining section from the toilet-soap finishing line. The refining section consists of three- or five-roll mills, followed by twin-worm duplex vacuum plodders. Twin-worm models have larger capacity than their single worm counterpart, give more refining action, and can handle sticky products, thus eliminating bridging and minimizing surging or pulsating of the extruded product. Uneven extrusion rates, characteristic of synthetic products processed on regular single-worm plodders, create subsequent cutting and stamping difficulties, hindering continuous high-speed operations. The twin-worm plodders can also be used for toilet soaps and nonaerated dense synthetic laundry bars.

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