alkaline catalyst (NaOH, KOH, NaOMe), is added to dry, neutral, or low acid oil at 80°C., and the mixture is stirred for a half hour. After settling, an anhydrous, dark glycerine layer is drawn off, and the supernatant methyl esters are washed free of methanol and alkali. The glycerine layer contains soap, color bodies, and free methanol. The resultant esters are usually considerably lighter than the original fat and of approximately 98% purity. For the preparation of fractionated fatty acids the esters may then be distilled in a column of inexpensive carbon steel construction. The ester of desired composition, or the whole ester, is then saponified with alkali and acidified to yield the split fatty acids.

The process has the advantage of utilizing relatively inexpensive carbon steel equipment for most of the process, requires no high pressure steam or equipment, and produces an anhydrous glycerine. It has the disadvantage of a multistage process and, most important, high cost of operation because of the considerable volumes of chemicals required.

Alkali Hydrolysis

The use of direct alkali saponification of a fat, followed by acidification of the resultant soap, is still being used to split some fats, but the practice is limited to a few isolated fats and oils and in small volume.

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Spray Drying

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THE PRINCIPLE of spray-drying soaps and detergents is not new. In fact, the majority of installations today do not differ greatly from those in use 30 years ago. However new concepts of spray drying developed over the intervening years have been the cause of considerable confusion regarding tower types to suit specific requirements. There have been some disappointing installations through ignorance of fundamentals (inexperience regarding retail-market requirements) or through a desire to avoid costly patent litigation.

J. W. McCutcheon

For many years retail soap powders have been sold in a hollow bead form, which provides a large area of contact with the water for quick solution. Solid forms, such as ground powders, have greater density, tend to coagulate into a paste, and generally are difficult to dissolve. For certain industrial purposes however these are not objectionable, and towers for producing such products are available. Also available are spray towers for removing the heat of crystallization from washing powders, etc. These latter are often called cold towers to differentiate them from towers the primary function of which is evaporation. Another type of spray-drying tower is the vacuum type adapted for the production of toilet and other soaps where combined cooling and evaporation takes place. Scientifically all these spray towers deserve equal space. My purpose is best served however if space is devoted to the most economically important process, namely, the spray drying of a bead type of product.

The equipment may be divided into six parts as follows: the tower body; air circulation system including ducts, fans, inlet and outlet openings, and dust separators; heating system; slurry preparation and feed; product removal system; and controls, maintenance, and safety.

A FEW GENERAL considerations on tower operation are as follows: The detergent or soap compound, as a 65-70% solids slurry, is introduced through spray nozzles into the top of a large cylindrical vessel, where it meets a rising blast of hot air. This strips it of its moisture with a rapidity designed to cause the small particle of soap or detergent material to explode into a hollow bead. The hot air enters near the bottom of the tower and is drawn off at the top. Because the product passes against the current of air, such a tower is called a countercurrent tower. When the air flows in the reverse direction from top to bottom, the tower is known as concurrent tower.

Towers are made to operate in either manner. Generally a countercurrent tower is used when the density of the desired product has a medium to high value of, say, 0.4 to 0.6 while a concurrent tower is used when very light products are desired with a density range of 0.2 to 0.4. The high degree of puffiing in the concurrent tower is caused by the hottest air hitting the particles just after they leave the nozzle, yielding maximum particle disruption. In the countercurrent tower the evaporation occurs less explosively because the particle passes through a gradually rising temperature zone. The majority of present towers are countercurrent and yield products with moderate densities from 0.5 to 0.6.

The capacity of a tower depends on the temperature and volume of the air circulated, tower design efficiency, and product formulae. The following is an illustration of what is meant by product formula. A feed of 1,000 lbs/hr. of a 70% slurry will require the evaporation of 263 lbs./hr. of water to produce 737 lbs./hr. of product containing 5% moisture. The same feed will require only 222 lbs./hr. evaporation to produce 778 lbs./hr. of a 10% moisture product. The productive capacity of the tower is increased an additional 25% by this moderate change in product specifications.

If a 65% solid slurry is fed to the tower, productive capacity, based on the tower's ability to evaporate water and on a 5% moisture product, is cut 23%. The type of builder also exerts an influence. Silicates tend to decrease evaporation rates. Hydrating salts, such as soda ash, permit higher moisture content of the product for the same free-flowing characteristics. The production capacity of a tower therefore is greatly influenced by the formulae of the products being made. This is a factor over which the design engineer has no control. He generally specifies tower capacity either in terms of water evaporated per hour or in terms of a specific product made in a specific manner.

Tower design is a fairly constant factor which is difficult to evaluate except by the comparison of one tower with another. A small diameter tower, for example, will increase air velocity to a point where the product may start to pass over into the dust collection system. Too large a diameter may cause considerable air to pass around the slurry particles and permit excess heat losses. Generally speaking, an air velocity of 1 foot per second is acceptable.

Other factors include the number and position of the nozzles, tower insulation, and total free-fall of the particle through the heated zone, a factor governed by the tower height. The time involved in drying the particle is extremely important and should be somewhere between 3 and 5 seconds. A short tower requires excessive heat and air volume. Too high a tower greatly increases the capital cost.

Although tower design is an extremely important factor in determining capacity, it is primarily one of engineering and has a fixed value. The volume and temperature of the air passing through the tower is partly fixed by engineering design and partly controllable by the operators. If, for example, 10,000 c.f.m. of air are being introduced into the tower at 500°F. and is being removed at 200°F., we can calculate that approximately 2,800,000 BTU per hour is being used to convert water to vapor and to maintain the tower heat losses. Within reasonable limits and with the other variables fixed, the tower capacity may be taken as proportional to the product of air volume by the temperature. At 450°F., for example, the air volume would be stepped up to 11,000 c.f.m. to maintain equivalent drying conditions. However the product under these two sets of conditions would not be the same. Lowering of the temperature would tend to increase the density. Generally tower-operating conditions are set up for each product and are closely followed.

Now to discuss the individual tower parts. The body is generally of carbon steel for soap, or stainless steel or stainless clad steel for soap and detergent. Other materials have been used such as wood, tile, etc., but such materials are not to be recommended. The outside should be insulated to reduce heat loss and other unsatisfactory tower conditions. The tower may be constructed outside the plant building with a covered platform at the top for operational purposes. A 5-ton-per-hour plant might have tower dimensions of 25 ft. in diameter by 100 ft. high.

The hot air is circulated in a countercurrent tower by bringing it in near the bottom at a temperature of 350-500°F. in a plenum ring. The inlet parts are protected by an inside skirt, which deflects the air down so that a swirling motion is imparted to it. This is helpful in keeping a uniform motion of the air across the diameter. The vapor-laden, cooled air at 200–250°F. makes its exit through a duct at the top to the exhaust fan. Interposed between fan and tower are one or more cyclones plus a wet or dry filter, depending principally on the type of products being run. Many towers operate satisfactorily without any filter between cyclone and exhaust fan.

A dry filter is preferred over a wet one if possible since dust is easily consumed in the crutching operation. The collection of fines from the cyclone may be added to the product if a screening operation is employed, but generally it also is added directly to the crutcher mix for reblowing. Both fines and dust should not exceed 5% of production. Ducts should be of a diameter sufficient to handle the volumes required without unduly increasing the fan load. A 3 ft. diameter duct, for example, will handle up to 15,000 c.f.m. The best shape is circular, and the layout should be streamlined. Ducts removing low-temperature, moisture-laden air from the tower must be insulated to avoid condensation. In particular, automatic dumping filters may run into trouble by caking on the cloth.

The heating of the air is best done directly by drawing it through a gas- or oil-fired furnace by tower input fans. This means that, in starting up the furnace, the air must be by-passed through a stack until all soot is burned off. Control of air temperature is obtained by an automatically regulated damper connected to an air duct by-passing the furnace. In sizing the furnace, consideration must first be given to the heat requirements for evaporation at maximum-rated tower capacity. To this must be added tower and furnace heat losses plus a suitable margin of safety.

Slurry preparation includes a pair of crutchers operated alternately, an agitated feed tank, a low-pressure pump, heat exchanger, deaerator, high-pressure pump, and nozzles. Screens should be located in the slurry lines to protect nozzles from clogging. One difficulty may be caused by the hydration of the polyphosphates during prolonged crutching to give small granular lumps that can cause a great deal of nozzle difficulty. The use of a heat exchanger in the slurry line may or may not be necessary, but the slurry must reach the nozzles at a controlled temperature for uniform blowing conditions. For highly puffed products this temperature may well exceed 220°F. Soap does not require deaeration, but detergent products usually do.

DEAERATION may be effected by vacuum equipment, centrifuge, or other means and is done just prior to the low-pressure pump which should deliver slurry to the high-pressure pump at 50 p.s.i. or so. The high-pressure pump provides the necessary pressure at the nozzles and operates over a variable pressure range from 500 to 2,000 p.s.i., depending on the product being blown. The nozzles must be placed in the tower in a definite pattern to utilize the full diameter without throwing the slurry on the sides. There is variety of ideas on how this should be done. Some towers of large diameters will have 20 or more nozzles spaced in geometric pattern over the top area. Others may have one or two only. Nozzle orifices are subject to wear and must be replaced frequently. Stainless nozzles with tungsten carbide inserts are satisfactory.

The best give a swirling motion to the slurry stream just before it reaches the orifice. Orifice openings vary from about 1/16 to 3/16 in. in diameter. The small orifice sizes give fine, dense particles. The larger orifices give more puffed products. Viscosity of the slurry and the pressure also affect product particle size and density, and these two factors must be kept under close control. With high pressures, such as 2,000 p.s.i., the air temperature may be lowered to 350°F. to yield a dense, fine powder. With lower pressures of, say, 800 p.s.i. and a temperature of 450°F. the product may be a light fluffy bead. All factors affecting the product however must be taken into consideration before nozzle conditions can be properly assessed. Some installations, without high-pressure pumps, use auxiliary air or steam to improve spray conditions. If such a system is used, it should be controlled carefully to avoid pulsations at the nozzles. The use of such aids is to be avoided in general.

The product falls into the cone bottom of the tower, where it is fed through a rotary feed valve to a conveyor belt or an air lift, from which it passes to storage. Perfume is frequently atomized into the product at this point. If an air lift is used, the size of the duct must be large enough to prevent breakdown of the product and yet small enough to keep the suction equipment economically sized. Such ducts will run about 6 in. in diameter for 1,000 lbs./hr. product transfer. Screening may or may not be used, and a dry dust collector may be inserted between the cyclone separator and the exhaust fan. An air lift has the advantage over a conveyor system in causing less breakdown of beaded products.

CONSTRUCTION MATERIALS must be chosen according to the products and service. With detergents, stainless or other noncorrosive metal is necessary at the points where the product is in moist contact with the metal. Air ducts need not be stainless but must be well insulated to prevent heat loss or moisture condensation. If stainless is used at all, it should be used on the exhaust ducts. The bearings on the intake fan should receive special cooling consideration. Temperature indicators should be used at the furnace, air inlet, and air outlet and on the slurry. Pressure indicators should be placed on the tower and on exit lines from the low- and high-pressure pumps. To avoid difficulties in tower-pressure variation large explosion doors should be installed near the top of the tower and fastened in place by shear bolts.

Another important safety measure is furnace control should the flame go out. This is handled by special automatic equipment. The inside of the tower body should be kept very clean, particularly when blowing detergents which are more heat-sensitive than soap. The tower body should be designed with smooth internal surfaces, and provision should be made to have some internal washing device not only for the rapid quenching of fires but also for regularly scheduled cleaning.

Not all spray drying towers for soaps and detergents are built along these lines. One tower, for example, was of an inverted V shape with the nozzle located in the middle upper section for horizontal blowing. The particles traveled in a parabolic curve and were collected on a conveyor belt. The air was exhausted through ducts along the upper section and was heated indirectly by steam pipes running along the base. The formula contained a good percentage of highly crystallizable salts, such as soda ash, and the particles were not true puffed beads but more nearly resembled miniature "jacks." The tower was producing a built soap product having a real soap content above 50%, therefore it could not be called a cold tower.

This latter depends almost entirely on the crystallization of salts in the formula as the product falls through the chamber. Generally such products are only partically crystallized by the process and must be allowed to stand a day or so in buggies to complete the process. A tower of this type can be made of almost any material because of the low temperatures employed and may be of simple construction.

Another type of spray dryer in the hot-tower class is that which is designed to produce a fine, dense product without regard to bead formation. One of the best known is the Kestner tower, where the slurry falls on a rotating disc and is centrifuged off as a very fine spray. This most nearly resembles the bead type of tower in principle and construction. The tower is squat and wide and can be constructed at less cost than the bead type. No high-pressure pumps are required, and there are no nozzles to clog. The product is unpuffed, fine, and generally dusty, characteristics which have been objected to by industrial users. This type can be converted to the bead type only at consideratble expense, largely through increasing the tower height. Then there are spray-drying towers where nozzles are used, and the entire product and air are swept from the tower and separated in the cyclone.

Spray dryers have been designed principally for various drying jobs, for which the drying of soaps and detergents is only incidental. Manufacturers of these towers include Bowen Engineering; Proctor Schwartz; Miro Atomizer Ltd., England; Swenson; Meccaniche Modern, Italy; Kestner, England; etc.

VACUUM-TYPE SPRAY DRYERS are being introduced by G. Mazzoni, S.p.A., for toilet soap manufacture. This unit is very small, about 5 ft. in diameter by 8 ft. tall. The soap is sprayed into the top, and partial evaporation of the moisture takes place at temperatures controlled by the heat of the incoming soap. The product is continuously scraped from the walls of the chamber and fed to a plodder for extrusion in bar form.

In summary, spray drying of soaps and detergents in bead form has been discussed at some length, concluding with a variety of spray systems designed principally for nonpuffed products and suitable for industrial use. The Mazzoni vacuum spray-dryer for toilet soap is mentioned.

Recent Advances in the Analysis of Soaps and Synthetic Detergents

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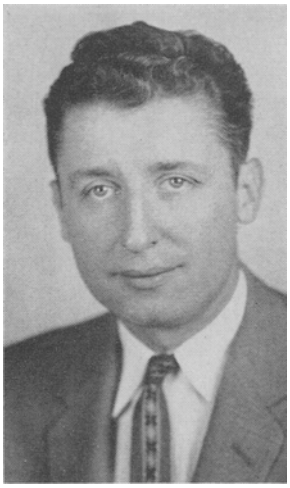
The methods of analysis for soaps and synthetic detergents in this paper are divided according to the various existing types of these materials. The methods to be discussed will include qualitative and quantitative approaches and will also include methods of separation. Whenever possible, the quantitative methods will range from the assay of the relatively pure soap or syndet to methods for determining trace quantities.


Sidney Siggia

The methods described in this paper include some not generally used for the determination of surfactants. These apply to classes of chemical compounds of which the surfactants are members. For example, existing methods for the analysis of carboxylate salts automatically can be applied to soaps; methods for tertiary and quaternary amine salts can be applied to many cationics; methods for hydroxy compounds and polyglycol ethers and esters can be applied to many nonionics.

General Methods

Those described in this section are not specific for any surfactant type and are generally applied to unknown samples for characterizing the soap or detergent present, or they are methods which are generally applicable to all types of surfactants.

Wurtzschmitt (58) describes a scheme of analysis for characterizing surface-active agents, which uses elemental and functional group analyses for generally classifying the materials and employs the preparation and isolation of derivatives for further clarification of structure and composition. It is possible to estimate the amounts of certain agents by isolation and then by determination.

Gilby and Hodgson (14, 15) also describe a scheme for characterizing surfactants. This scheme is based mainly on the Lassaigne test for certain elements. Briefly the scheme is as follows.

a) The presence of carbon, hydrogen, and oxygen alone generally signifies a soap or a nonionic.
b) Sulfur generally indicates a sulfate or sulfonate.
c) Sulfur and nitrogen indicates fatty acid esters of sulfoacetamides.
d) Nitrogen indicates cationics.

Yet another series of characterization tests is given (3), which can be summarized as follows.

a) Methylene blue associates with sulfated and sulfonated surfactants, making them soluble in chloroform. If a blue color develops in the chloroform layer after addition of methylene blue, then these types of anionics are likely to be present.
b) A reagent composed of ammonium thiocyanate and cobalt nitrate should give a colored solution that remains colored in the presence of a nonionic.
c) Quaternary ammonium salts cause bromphenol blue to go into a chloroform layer.

The latter includes methods for detecting various ingredients in surfactant formulations and also includes some quantitative approaches that might be used for determining the active ingredients.

Kortland and Dammers (19) describe a rather intricate system of qualitative and quantitative methods for analyzing anionics, cationics, and nonionics. Balthazar (7, 8) also describes a scheme for the same general purpose.

The most conclusive and most rapid method available today for identifying a surfactant is infrared absorption. The American Society for Testing Materials (31) has published infrared absorption curves for many types of known surfactants. Therefore the first step to be taken in an identification is to obtain an infrared absorption curve on an unknown and to compare this with the curves for the known surfactants. If a matching curve is found, then the identification is complete with no further work required. If no matching curve is found, then the problem of identification becomes more complex though infrared absorption can help in these cases as well. Infrared will differentiate the aromatic from the aliphatic type of compounds. Also it will identify the functional groups on the molecules as a great aid in an identification. Applications of infrared absorption to the quantitative analysis of organic compounds are discussed by Siggia and Stolten (48).

Ultraviolet absorption does not have the range of applicability as infrared absorption. However the ultraviolet approach has the advantage of being less bothered by interferences. Most compounds absorb in the infrared region of the spectrum whereas a much lower percentage absorb in the ultraviolet region. Hence infrared analysis may require some resolution of components in a mixture before the analysis is applied; analysis *via* ultraviolet requires resolution of