53. (a) McBain, J. W., Elford, W. J., and Vold, R. D., J. Soc. Chem. Ind., *59*, 243 (1930); (b) Ferguson, R. H., and Richardson, A. S., *24*, 1329 (1932); (c) Vold, R. D., and Ferguson, R. H., J. Am. Chem. Soc., *60*, 2066 (1938).
54. Maclennan, K., J. Soc. Chem. Ind., *42*, 393T (1923).
55. McBain, J. W., Gardiner, K., and Vold, R. D., Ind. Eng. Chem., *36*, 808 (1944); McBain, J. W., Thorburn, R. C., and McGee, C. G., Oil and Soap *21*, 227 (1944).
56. Warwicke, J., Textile Colorist, *61*, 315-7, 378 (1939); Soap and Sanitary Chemicals *15*, 58 (August, 1939).
57. Smith, E. L., Chem. and Ind., *58*, 87 (1939).
58. Booth, J. H. W., Chem. and Ind., *56*, 1120 (1937).
59. Merrill, R. C., and Getty, R., J. Phys. and Colloid Chem., *52*, 167 (1948).
60. Merrill, R. C., unpublished data.
61. Edeler, A., Ind. Eng. Chem., *17*, 196 (1925).

Continuous Fat Splitting Plants Using the Colgate-Emery Process*

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A CONTINUOUS high temperature fat splitting process, employing countercurrent reaction in a pressure tower with internal heat exchange, has been developed and carried through pilot plant investigation to successful commercial operation. This method, known as the Colgate-Emery Fat Splitting Process, gives splitting efficiencies of about 98%, producing acids which can generally be bleached to a color equal to or better than that of the original fat. Because of savings in steam, since no catalyst is needed, the new process shows considerable economy over the old Twitchell method.

The earliest attempts to split fats were in soap making, where the fatty acids appear as sodium or potassium soaps and the glycerine is either left in the soap or is separated by salting-out. Fatty acids are readily made from soap by acidulation, but the overall process is indirect and costly in chemicals. Chevreul, in 1813, discovered that fat is a regular chemical compound of glycerine and one, two, or three fatty acids. He also discovered that fat can be separated into these parts by causing it to unite with water. In 1853 Tilghman (1) discovered that the reaction of fat and water, to form fatty acids and glycerine, can be carried out by mixing the fat with water and then subjecting the mixture to a high degree of heat while the pressure is maintained sufficiently high to prevent vaporization of the water. This inventor was in advance of his time, as materials of construction and techniques of operation for the required high temperature and high pressure conditions were then unknown. Twitchell, in 1890, took a forward stride when he devised a relatively simple method for producing fatty acids and glycerine directly from fats. This is an atmospheric pressure boiling method employing a reagent or catalyst to speed up the hydrolysis. Twitchell's method is widely used and will not be replaced entirely for many years because it is still a good process for certain types of splitting. Batch autoclave splitting at 100 to 150 psi with lime, magnesia, or zinc catalyst, has been widely used, especially in Europe, while the batch process at around 400 psi without catalyst has found limited popularity. Continuous countercurrent high temperature splitting, as carried out by Procter and Gamble (2, 3) and more recently in novel form by Colgate-Palmolive-Peet and Emery Industries (4, 5) constitutes a marked improvement over Twitchell and is finding wide immediate acceptance, particularly for large scale operation. In the Colgate-Emery Process fat and water react countercurrently in a column at about 500 degrees Fahrenheit and about 725 psi. Heat exchange between fatty acid and water takes place in the top portion of the column and between fat and sweet water in the bottom part.

The fatty acid industry originated early in the last century and first produced stearic acid for candles. The present day stearic and oleic acid industry, based primarily on animal fats, has developed from this primitive origin and now produces materials which find their way into a wide variety of uses. Moreover the field for fatty acid from other sources such as vegetable oils and hydrogenated oils is steadily expanding. Because of the advantages of countercurrent fat splitting as a step in soap making, indications are that future expansion of the soap industry will continue in this direction.

Data on hydrolysis of many fats in a pilot countercurrent splitting plant built by Colgate-Palmolive-Peet have been published by Allen and coworkers (6). One of the present authors proposed an improvement of the original process consisting of the addition of internal heat exchange (5), of which the equipment details were reported in the last reference. A number of runs were made in the modified unit, and sufficient data were obtained to design a commercial plant.

The selection of the optimum temperature-pressure operating range called for a careful analysis of many factors. The higher the temperature (and therefore the pressure) the faster the reaction rate. The higher the temperature, the greater the solubility of water in fat, which is desirable until the amount becomes so great that the amount of water in the column cuts down on the tower capacity. The higher the pressure, the greater the cost of equipment per unit of volume but with increased reaction rate the volume of the splitting zone can be smaller. If the temperature is too high, it may have a deleterious effect on certain fats and may in fact make counterflow impossible by causing complete miscibility. A temperature of around 500°F. was selected as a practical compromise, and a maximum operating pressure of 725 lbs. was chosen to provide a suitable excess to prevent boiling (vapor pressure of water at 500°F. is 669 psi).

The first commercial plant, having a design capacity of 3,000 lbs. of feed per hour, intended primarily for low-grade fats, was built at Cincinnati, Ohio, by Emery Industries and has been in operation since

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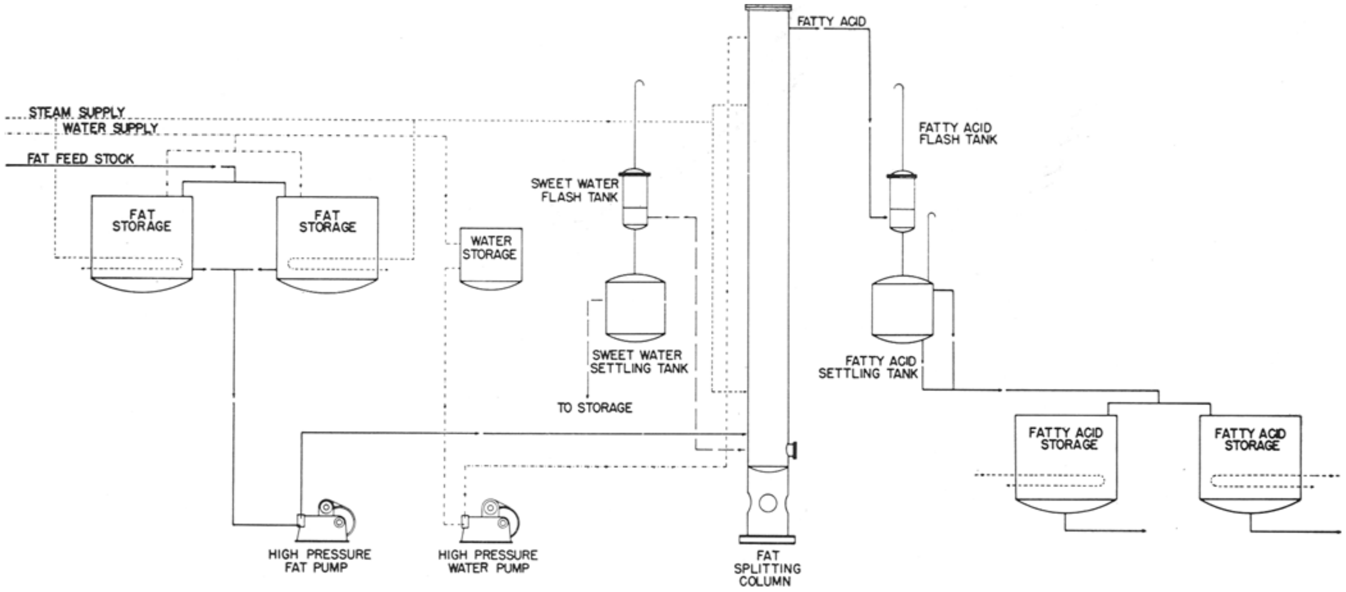

FIG. 1. The operation of the Colgate-Emery Process is shown by this simplified flow sheet.

April, 1947. The second commercial unit, having a capacity of 5,000 pounds per hour, is now being built by Blaw-Knox for Swift and Company at Hammond, Indiana. It is expected that this plant will go into operation within the next few months. The commercial operation of this splitting process is shown on the flow sheet and is described below (Fig. 1).

The fat is pumped from tank cars, storage, or purification into alternate feed tanks. From these it is charged into the bottom of the splitting column by a high pressure plunger type feed pump. It enters through a sparge ring which breaks the fat into small droplets and, as it becomes heated, it rises through the sweet water accumulating section where its temperature is increased by direct contact with the sweet water. After passing through the fat-water interface, the fat is further heated by direct sparged steam to about 500 degrees Fahrenheit. Hydrolysis takes place as the fat (the continuous phase) passes upward through the tower. The column is heavily insulated to minimize heat loss. The small amount of heat needed to maintain the contents at the splitting temperature may be supplied either by direct high pressure steam admitted to the central portion of the tower, or by external electric strip heaters.

Process water is withdrawn from its tank and is charged into the top of the splitting tower through a sparge ring by a high-pressure plunger-type pump. The water is heated by direct contact with the fatty acid in the internal tray-type heat exchanger and is then re-dispersed by a distributor plate. The additional heat required to bring the water to 500 degrees Fahrenheit is supplied by direct steam. The water, in the form of fine droplets, falls through the column of fatty acid and fat and the sweet water is accumulated at the bottom of the tower prior to discharge by an automatic interface controller.

The fatty acid is discharged from the top of the tower through a back-pressure control valve which maintains the column at working pressure. The column is completely filled with liquids and vaporization of water is prevented by maintaining the pressure in excess of the vapor pressure of water at the operating temperature. The fatty acid passes to a flash tank where the temperature is dropped by evaporating part or all of the water which it carries and then to a settling tank which removes the remainder of the water. The settling tank serves also as a surge tank for recycling the product in starting. From there it goes to heated fatty acid storage tanks.

The sweet water goes to a flash tank and then to a settling tank where small amounts of fat and dirt are removed by skimming. The sweet water is now ready, after a light lime treatment, to be sent to the glycerine concentrator. There is no severe scaling of the tubes with calcium sulfate as is the case with Twitchell split sweet water. For the same reason the finished glycerine has an exceptionally low ash content.

Untreated Cincinnati water is used in the Emery plant as process water. If color is of extreme importance, as in the case of high quality soaps, it may be advantageous to de-aerate the water.

High pressure steam, at 800 psi, is supplied by a steam generator or a steam compressor. In the Emery plant steam is supplied by a small high pressure boiler, using condensate from the steam chests of the glycerine concentrators as feed water. Treated city water would also be suitable. If there is already available a steam supply at a pressure lower than 800 psi, it can be boosted to the required pressure by a steam compressor. It is particularly advantageous to use this system when steam is being produced in the main plant boiler house at 300 to 400 psi.

Plant experience has shown that almost any type of fat that is free of suspended matter, including still residues, vegetable oil foots, etc., can be handled in the equipment. Lowest grade fats are advantageously given an acid boil prior to splitting, but this is not a critical requirement as in Twitchell splitting because in the present process impurities do not reduce the rate of hydrolysis. Where color is of extreme importance, it may be desirable to de-aerate the fat before charging it into the column.

The fatty acids can be used as produced for many types of products because of their light color and high degree of split. If high grade fats are used to

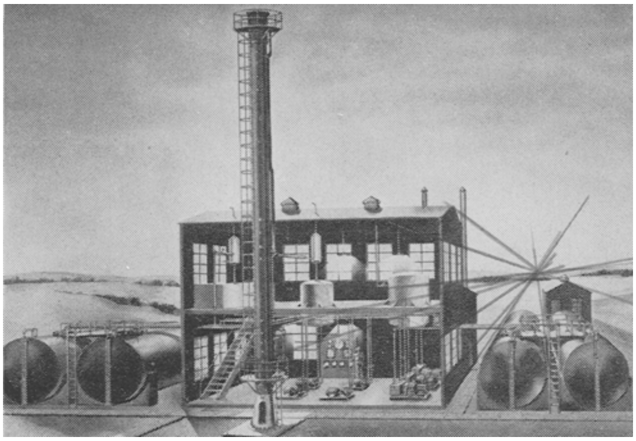

FIG. 2. This artist's picture shows a typical layout and the relation of the individual equipment items.

feed the splitting plant, it may be possible to produce satisfactory soap without distilling the fatty acids. Where highest purity is desired or if the feed stocks are of low grade, distillation is necessary. The product is clean and does not require neutralization or washing prior to distillation. While the fatty acid is in most cases darker than the original oil, it can in general be bleached to a color equal to or lighter than that of the original.

The first commercial plant of this type was designed to handle 3,000 pounds per hour as compared with 70 pounds per hour for the pilot plant. This meant stepping up in the ratio about 40 to 1, which introduced a number of interesting problems. One was the matter of column size and proportions. Would a larger tower split as much fat per cubic foot of volume as a small one? Would the water droplets tend to channel in a commercial tower? Should the tower capacity be stepped up by increasing the cross sectional area or the height, or both? The final answer to this was a compromise; the tower volume was made proportional to the capacity; most of the increase was taken on the cross sectional area, but part of it was taken in the tower height. The capacity and efficiency of the first commercial splitting tower indicated that the method of sizing and proportioning was approximately correct.

The distributing devices for admitting fat, water, and steam required considerable study for the commercial plant. Moreover the economic operation of the plant depends to a great extent on the efficiency of the internal heat exchange. The design of the commercial unit from the pilot plant data depended more on practical judgment than theoretical analysis. Results of plant runs indicate that some revision of the upper heat exchange zone will be repaid in steam economy, while the lower heat exchange zone came fully up to expectations.

One of the important practical problems in the designing of a commercial fat splitting plant is the selection of suitable materials of construction. The splitting column operates at 500 degrees Fahrenheit in contact with water and fatty acids and must give many years of safe operation at 725 psi. The material selected must give a long service life, must be commercially obtainable, and must be suitable for shop fabrication. The materials which best fulfill these requirements are Inconel an[illegible] Type 316 test samples usually s[illegible] sion tests, but the corrosion resistanc[illegible] is affected by the forming, welding, and [illegible] ment to which it was subjected in the shop; a[illegible] test samples cannot readily be prepared in a manner simulating shop treatment. Low carbon content 316 (below .05% carbon) is not as susceptible to damage from fabricating and heat treatment and may prove to be a superior construction material for splitting columns. At present, however, this alloy is difficult to obtain in the form needed for fabrication.

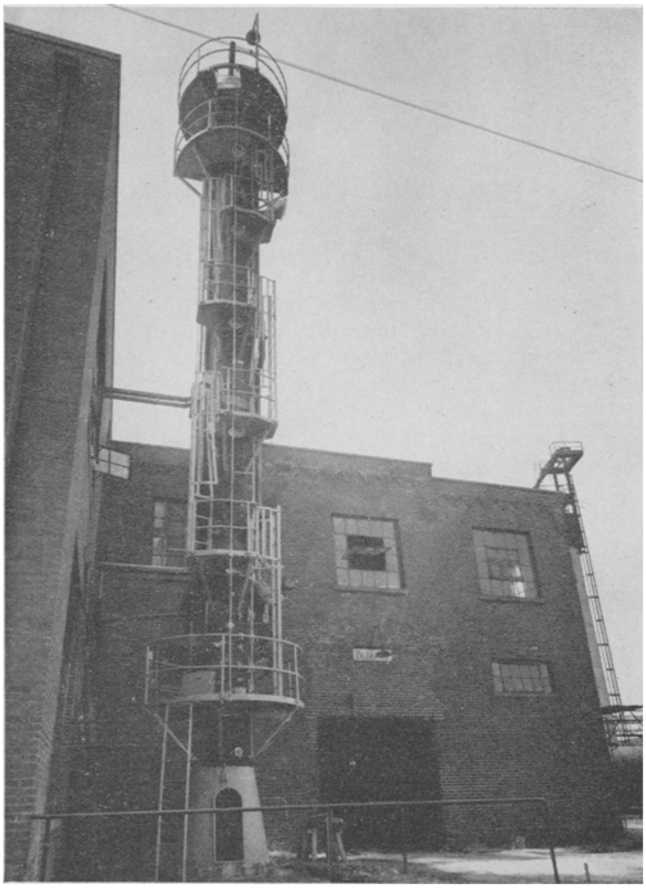

FIG. 3. The splitting column is about 60 feet high and is lined with corrosion resistant alloy.

Inconel is attacked more than Type 316 by fatty acids and water at high temperature but still has a low enough corrosion rate to indicate long life. Inconel does not depend on heat treatment for its corrosion resistance and in addition can be obtained and fabricated in the form of Inconel-clad steel. Inconel "B," which has a chromium content higher than the standard alloy, has shown improved resistance to high temperature fatty acids and is worthy of consideration.

The fat storage tanks can be fabricated of carbon steel, stainless steel, or aluminum, depending upon the type of fats used. Blow-down tanks, settling tanks, and fatty acid storage tanks are constructed of stainless steel, Inconel or aluminum, depending upon the service conditions. All parts of the high pressure charging pumps in contact with the materials being handled should be of stainless steel. The

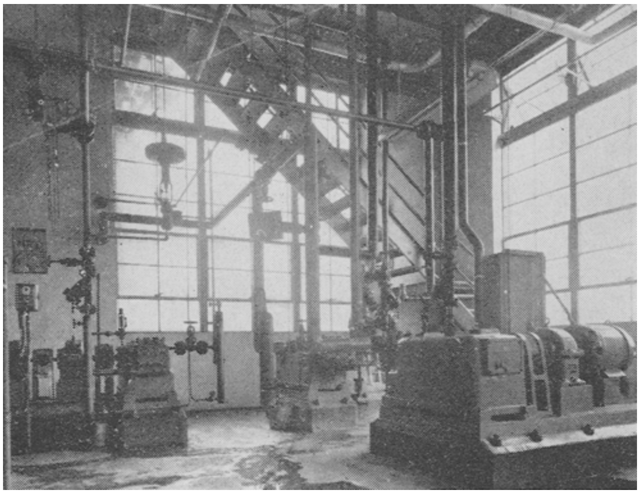

FIG. 4. A spare pump, to be used in place of either feed pump, insures uninterrupted operation.

process piping in contact with the fatty acids should be of Inconel or Type 316 stainless.

The Emery plant has processed principally low grade animal fat. In addition to this, a number of other common fats have been treated successfully, including hydrogenated tallow, hydrogenated fish oil, coconut oil, coconut foots, cottonseed-soya foots, as well as still residues. We hope to make data from some of these runs the subject of a future paper.

TABLE I

Data From Runs on Low Grade Animal Fat; Acid Boiled "Distillation Mix"

Operation	(a)	(b)
Free fatty acid of feed, as oleic	39%	28%
Throughput, lbs. per hr	3450	4500
Process water rate, lbs. per hr	1690	1800
Free fatty acid of product, as oleic	99.3%	98.1%
Glycerine in sweet water	12.0%	16.7%
Operating Requirement of Colgate-Emery Process per 1,000 lbs. feed		
800-lb. steam*	184 lbs.	154 lbs.
Electricity (power and light)	5.3 KWH	4.1 KWH
Process water	485 lbs.	400 lbs.
Labor (1 operator)	0.29 man-hr.	0.22 man-hr.
Comparison, Plant Two-Boil Twitchell Process Operation on Similar Mix		
Product FFA as oleic	96	97
Glycerine in sweet water	11%	13%

*Does not include low pressure steam used for tank heating, etc.

Some data from plant operations on low grade animal fats are given in Table I. The mix run in operation (a) was a mixture of No. 1 tallow, Special Tallow, Diamond S Tallow, yellow grease stearine, animal grease, extracted grease, etc., having a high initial free fatty acid. The run is comparable with the Emery Twitchell operation on the basis of approximately equal glycerine concentration, and shows over 3% more free fatty acid in the product.

Operation (b) was a run at nearly the maximum capacity of the feed pumps, using a mix of mostly animal grease, containing some animal tallow, etc., with a lower initial free fatty acid than the mix used in (a). The data show that even at a high feed rate the free fatty acid of the plant Twitchell process is exceeded, with considerably higher glycerine concentration in this case. The surprisingly small drop in free fatty acid at the higher rate is noteworthy, and the significance is under investigation.

Utility and labor requirements corresponding to the respective operating conditions are included in the table and are based on operation exclusive of starting and stopping. The electricity and labor items are constant hourly charges as one man operates the plant and the feed pumps run at constant load. These are reduced therefore in proportion as the throughput is increased. In addition to the high pressure steam requirement shown, low pressure steam is used to melt the fat and heat it to 180-200°F.; to warm the water to approximately 100°F., and to keep the traced pumping lines warm. This is unmetered, and would naturally vary with storage and other conditions.

With regard to possible limitations of the process a limited amount of laboratory work on linseed oil indicates that the Iodine Value drops a little, but not to a serious degree. Fish oil, however, has an Iodine Value drop of about 20 to 40 points, and it may be that fish oil cannot be satisfactorily split by the standard process being discussed.

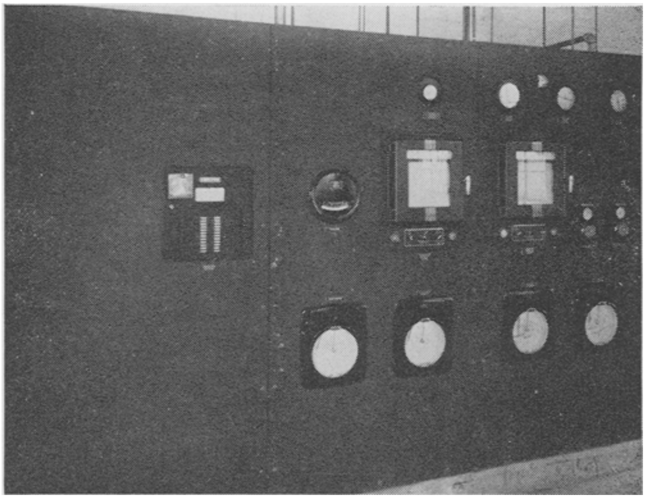

FIG. 5. The entire plant is controlled from a single instrument panel.

It has been found possible to switch from one type of feed stock to another with little or no intermixing. This is done by stopping the fat feed but continuing the water feed. As soon as all of the fatty acid has been discharged from the tower the new feed stock is started in. During this period the upper heat exchange section functions while fatty acid is discharged, and the lower one functions while the new fat is pumped in. About eight hours' time is lost and all of the fat from both feed stocks during the switch-over is satisfactorily split.

Some advantages of the process described in this paper are:

1. The process can be operated at high rates and with high degrees of split without the use of catalyst. The plant capacity can be further increased by using a small amount of catalyst and in certain cases this may be a reasonable thing to do.
2. Compared with the Twitchell process, treatment of the charging stock is much less critical. With high grade fats an acid boil is not required and it is only necessary to remove the suspended impurities.
3. By this process a split of 98% can be obtained.
4. The fatty acid product is considerably lighter in color than that obtained by the Twitchell splitting of the same

grade of fat and is free from unsaponifiables introduced by the Twitchell reagent. With high grade feed stocks, it is possible to make a good grade of fatty acid without distillation, which can be used for soap and many products. By a suitable bleach the product can be made generally lighter in color than the original fat.

5. With higher grade fats, the glycerine concentration in the sweet water can be 13 to 18%, or in general more concentrated than obtained by the Twitchell method. A light lime treatment is all that is required to coagulate impurities and the concentrated glycerine is practically ash free.
6. The process has exceptional heat economy, splitting more than five pounds of fat for every pound of high pressure steam used.
7. All of the inherent advantages of a continuous and countercurrent process are realized, including ease of control and operation, uniformity of product, low labor cost, high through-put, low uniform steam and power consumption, small space requirement, and low interprocess inventory.
8. The overall cost of splitting is less than by usual methods.

The process carries out in a modern continuous way an operation which has for almost a century been handled by a batch method. Since it is a continuous process it fits well with any other continuous process, for example those processes involving solvent extraction prior to splitting and distillation, solvent separation [Emersol process (7)], and neutralization (for the manufacture of soap) following splitting.

Fatty acids are basic chemical building blocks and can be used as intermediates for a multitude of products. By fractional distillation and solvent crystallization individual fatty acids can be prepared which can be used as starting materials to make specific chemical compounds.

The standard process can be modified to meet specific requirements. In the case of certain special products, such as drying oils, it may be desirable to operate at a somewhat lower temperature and pressure, even though the column size must of necessity be larger. For plants having capacities less than about 3,000 lbs. per hour, it may be good engineering to use a lower pressure, thereby having a column size which can be more easily fabricated, and will afford access for inspection of the interior. By the use of a small amount of catalyst the through-put can be increased greatly or the operating temperature may be lowered.

In addition to its use in fat splitting, the process may be of general interest for carrying out certain types of chemical reactions. Any two immiscible (or partially miscible) liquids which react reversibly at elevated temperature and pressure might be handled in the same type of plant. As an example, the hydrolysis of chlorobenzenes with aqueous caustic to form phenols might be carried out in this type of apparatus.

The problems involved in the commercialization of this method of operation have been successfully solved. Commercial plant results have demonstrated that most of the commercial fats can be split to a high degree and at a low cost. The process is finding wide application for purposes such as fatty acid manufacture and soap making. The general processing method and plant equipment may be adapted to other chemical reactions between two immiscible liquids.

REFERENCES

1. Tilghman—Patent No. 11,766.
2. Procter and Gamble—"Continuous Process for Soap," by Gordon W. McBride, Chemical Engineering—April, 1947.
3. Mills, V.—U. S. Patent 2,156,863 (May 2, 1939).
4. Ittner, M. H.—U. S. Patent Rei. 22,006 (Jan. 13, 1942).
5. Brown, A. C.—Patents applied for in U. S. and foreign countries.
6. Allen, H. D., Kline, W. A., Lawrence, E. A., Arrowsmith, C. J., and Marsel, C., Chemical Engineering Progress *43*, 459 (1947).
7. Kistler, R. E., Muckerheide, V. J., Myers, L. D.—"The Commercial Solvent Separation of Fatty Acids"—Oil and Soap, May 1946, Vol. XXIII, No. 5, pp. 146 to 150.

Preparation and Properties of Cottonseed Protein Dispersions[1]

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THE utilization of vegetable proteins in the manufacture of fibers, films, adhesives, paper and textile sizes, cold water paints, and related products is dependent on various physical properties of the proteins *per se* and of their aqueous dispersions. A number of these properties, *e.g.*, dispersibility in various media and the viscosity characteristics of concentrated dispersions have been investigated in the case of soybean and peanut proteins. Similar but less extensive investigations have been reported for cottonseed proteins. These investigations include the factors which influence the peptization of protein in solvent-extracted and hydraulic-pressed cottonseed meal (1), cottonseed proteins prepared under different processing conditions (2), the peptization of cottonseed proteins by various acids and bases (3, 4), the pigments of cottonseed (5, 6, 7, 8, 9), methods for separating the pigment glands from cottonseed (8, 10), and the viscosity characteristics of relatively concentrated dispersions of cottonseed protein (11).

The last mentioned article is also concerned with the difficulties of avoiding gel formation during the preparation of cottonseed protein dispersions in high protein concentration and in preparing dispersions having tacky and viscous characteristics.

The industrial utilization of cottonseed meal and proteins has heretofore been retarded because the extracted proteins are not appreciably dispersible in aqueous alkali solutions below pH 11.7. The present report is concerned with an investigation of methods for preparing nongelling dispersions of cottonseed

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