# **Fat Splitting**

# **O. J. ACKELSBERG, E. F. Drew and Company, Inc., Boonton, New Jersey**

THE NATURALLY-OCCURRING vegetable and animal oils consist mainly of the glycerol tri-esters of the fatty acids and are contaminated with many oils consist mainly of the glycerol tri-esters of the fatty acids and are contaminated with many minor constituents in varying degree, such as vitamins, phosphatides, color bodies, sterols, free fatty acids, etc. We shall be concerned with the splitting of



the glyceride esters of the fatty acids.

The use of fatty acids as such, in place of the natural fats and oils, has been steadily increasing, and there have been corresponding improvements in the splitting of the latter to produce acids of higher purity and at lower cost. Subsequent distillation, fraetionation, crystallization, etc., of the crude fatty acids have increased their applicability and have permitted the economic utilization of lower grade, lower cost feed stocks, such as acidulated soap-stocks.

The term hydrolysis, as

applied to fats and oils, refers ideally to the chemical addition of three moles of water to one mole of triglyceride to produce one mole of glycerine and three moles of fatty acid. This is the ideal reaction and is never fully attained in practice but is only approached asymptotically.

The glycerol esters of fatty acids have been split with alkali to produce soap for at least 2,000 years. The Roman, Pliny, recorded the treatment of goat's tallow with causticized beech ashes, and earlier Egyptian records reported the use of lime grease. It was not until the early part of the 19th century, though, that the French chemist, Chevreul, proved the basic structure of fats and oils as the triglycerides.

## **Chemistry and Kinetics**

The hydrolysis of triglycerides is a reversible reaction in equilibrium with esterification.

Lascaray (18, 19), Sturzenegger and Sturm (32), Mills and McClain (24), Yonese (37, 38, 39, 41), Ueno (34), and Hartman (10, 11) among many others have agreed that the reaction proceeds stepwise from triglyceride to diglyceride to monoglyeeride to glycerine and as an homogeneous first-order reaction in the oil phase. Yonese (6) isolated by molecular distillation the mono-, and diglycerides formed during successive stages of hydrolysis of coconut oil in an autoclave.

Mills and McClain (24) determined that the glyc. erine content of the unsplit fat sampled during the autoclave splitting of coconut oil and tallow almost doubled until a maximum was reached at approximately 90% of hydrolysis. The monoglyceride content then remained constant until the maximum theoretical split was obtained, indicating that the relative rates of hydrolysis of the mono- and diglyeerides are about equal at that stage. Mueller and Holt (28) reported that, during the Twitehell splitting of coconut oil, the diglyceride content rose sharply from 7.6% to 19.5% in a little under four hours before dropping in  $17\frac{3}{4}$  hours to  $7.08\%$  after the first boil. In one hour on second boil the diglyceride content dropped to 1.59% and disappeared completely in the third boil. The monoglycerides during the same period rose from  $0.58\%$  to  $7.04\%$  in  $6\frac{3}{4}$  hours and fell to 0.8% at the end.

The hydrolysis is a first-order reaction in the oil phase. The heterogeneous reaction between water and oil at the interface is of minor importance in the hydrolysis as compared with the homogeneous reaction with the oil of the water dissolved in the oil. Since water has a limited solubility in oil at low temperatures, the reaction proceeds very slowly at the start, and a demonstrable induction period has been reported (18, 19, 32). During this early stage, both in Twitchell and high-pressure splitting, an emulsion may be formed. With the liberation of free fatty acids in the oil phase the emulsions begin to disappear, the reaction accelerates and proceeds at an almost constant rate for a given temperature. This change from the slower heterogeneous reaction to the more rapid homogeneous reaction correlates closely with the increase of fatty acid content and the water content of the oil phase. This results from the increased solubility of water in fatty acids (Figure 1). The solubility of water in fatty acids increases markedly with the rise of temperature, and the rate of reaction accordingly increases. Sturzenegger and Sturm (32) have presented experimental data on the effect of temperature on the rate of autoclave hydrolysis of tallow, coconut oil and peanut oil, using water-to-oil ratios of 70%, 90%, and 50%, respectively (Table I). Hydrolysis at  $225^{\circ}$ C. is very slow but is almost instantaneous at  $280^{\circ}$ C. Lascaray (18) has tabulated the rates of hydrolysis at lower temperatures (Table II), using the reaction rate at  $150^{\circ}$ C. as unity and at a constant 60% water-to-oil ratio. The use of a catalyst which increases the solubility of water in the oil phase, such as ZnO or *MgO* (by forming metal soaps), markedly increases the rate of reaction.

A temperature coefficient for fat hydrolysis has been calculated by Lascaray (18, 19) and confirmed by Sturzenegger and Sturm (32). A temperature increase of  $10^{\circ}$ C. increases the rate of reaction by a factor of 1.2 to 1.5. Most homogeneous reactions exhibit temperature coefficients of  $2$  to 4. The hydroylsis reaction however is limited to the 1.2-1.5 range by the





Fig. 1. Solubility of water in fatty acids (24).

much slower physical process of diffusion. There is no measurable heat of reaction.

Sturzenegger and Sturm demonstrated that the degree of hydrolysis is not a function of temperature by obtaining approximately equal equilibrium endpoints at  $225^{\circ}$ ,  $240^{\circ}$ , and  $280^{\circ}$ ; the reaction velocities varied, of course, with temperature. These results may be seen in Figure 2, in which a water-fat ratio of 70–100 was used for beef tallow. All approach the same asymptote, although at different rates.

Since hydrolysis is an equilibrium reaction, the completeness of the reaction is a function of the concentration of glycerine left in the oil phase. This relationship has been clearly demonstrated by Lascaray  $(18, 19)$  and Mills and McClain  $(24)$ . Following the law of mass action, the equilibrium will shift towards greater or lesser hydrolysis as the concentration of glycerine in the oil phase falls or rises. Lascaray has evolved a simple formula for characterizing the degree of hydrolysis at equilibrium as a function of this glycerine concentration (This formula is valid only in systems where the glycerine concentration is less than  $20\%$ .):

 $H = 100 - 0.8$  G

where H is the degree of hydrolysis at equilibrium and G is the concentration of glycerine in the aqueous phase.



FIG. 2. Hydrolysis of beef tallow (32).

**TABLE II** Relative Rate of Hydrolysis at Different Temperatures

Temperature. °C.	Relative rate
	0 03
	2.4
	5.5
	13.9

This in turn is related to the ratio of water-to-oil used and to the rate of diffusion of glycerine from the oil phase to the water phase.

Mills and McClain (24) have shown that an oil in equilibrium with a glveerol-water solution will dissolve a glycerine-water solution of the same concentration. The ratio of water to oil is therefore the limiting factor for the reaction. The degree of completeness of reaction at equilibrium is independent of the temperature or catalyst used and is governed wholly by the ratio of water to oil used. The marked effect of



FIG. 3. Effect of percentage of glycerol on the point of equilibrium for coconut oil system  $(24)$ .

increasing the water to oil ratio is readily seen for this Twitchell reaction in Figure 4. It has also been determined that the rate of hydrolysis is independent<br>of the type of fat used. Mills and McClain found similar results for both tallow and coconut oil (24).

The data of Sturzenegger and Sturm (32) are tabu-





lated on the basis of mole ratios over an extremely large range for the hydrolysis of beef tallow. Sturzenegger and Sturm (32) have further shown conclusively that the degree of hydrolysis is independent of the reaction temperature. The data are shown in Table IV.

By lengthening the reaction time at lower temperatures to permit equilibrium to be attained, no difference can be seen.

The action of catalysts in increasing the velocity of the reaction is believed due (18) to two processes: increase of water solubility in oil and increase of ionization in the water dissolved in the oil. Both the sulfonic acids used for the Twitehell process and the



heavy metal oxides used for the high-pressure process have little effect at the start of the reaction until they have dissolved in the oil phase, at which time their action is marked. Both these materials are essentially insotubIe in neutral oil but are increasingly soluble in oil containing fatty acids. Sulfuric acid in Twitchelling is believed additionally to lower the solubility of the splitting agent in the aqueous phase, thereby increasing its solubility in the oil phase. This increases the hydrogen ion concentration in the fat phase where hydrolysis occurs.

Stirton *et al.* (31) have determined the phase distribution of splitting agent between oil and water in the TwitehelI-splitting of lard. They demonstrated (using sulfoxylyl-stearie acid) an inverse correlation between degree of hydrolysis and percentage of splitting-agent in the aqueous phase. At the start of hydrolysis the reagent is present 100% in the aqueous phase and, as it shifts to the oil phase, reaction progresses to 85% at 90% transfer.

Acid catalysts are in general of greater efficiency in catalyzing hydrolysis reactions and arc so used in the Twitchell process. They have been replaced in the high-pressure process by the basic oxides because of the corrosiveness of the former. Lasearay has investigated a series of these bases for catalytic activity in splitting tallow at  $185^{\circ}$ C. with 60% water and reports the results as follows, using pure water as unity :



The divalent metals are more active than the univalent metals (in the form of oxides).

In summary, fat hydrolysis is an equilibrium reaction in which the reaction velocity is a function only of temperature or catalyst, and in which the degree of completeness of reaction is a function of the concentration of glycerine in the water phase and therefore of the oil-water ratio.

#### **Processing**

The batch processes are more adaptable to the handling of either smaller lots of stock or of frequently varying types of stock. The installations are less expensive. The continuous processes, with higher initial cost, produce greater uniformity at a usually lower operating cost.

#### **Twitchell Process**

Acid-splitting of oils in the presence of sulfuric acid and a surface-active agent was first successfully developed by Ernst Twitchell (33) in 1898. This process is still the most widely used method throughout the world for the splitting of fats and oils, principally because of low initial cost and simplicity of installation and operation. It requires a minimum amount of expensive and technically-complicated equipment, making it particularly suitable for the simplest installations where, quite apart from initial costs, the availability of high pressure steam or technical manpower is limited.

It is a batch process conducted in open or vented, covered tanks of Monel, wood, or lead-lined construction. The fat is mixed with 25-50% of its own weight of water, 0.5 t0 1.5% of splitting reagent and usually 1% of sulfuric acid. Literally thousands of reagents have been proposed and used for the process. The early ones usually were the reaction product of the sulfation of a 50-50 mixture of oleic acid and an aromatic hydrocarbon, such as naphthalene or xylenc. Many of the compounds in use today are of the familiar alkylarylsulfonate types associated in our minds with synthetic detergents. Petroleum sulfonates are also widely accepted.

In the process the charge mixture is agitated with live sparge steam for from 12 to 24 hrs. and then is allowed to settle. Glycerine water is drawn off, and the supernatant fat is usually 80-85% split. The fat is usually then given a second boil with fresh water and sulfuric acid with no addition of catalyst; the latter remains in the oil phase from the first stage. A split of  $90-95\%$  may be obtained. If a higher degree

Composition	Sample No.							
		10	20	24	31	41	45	
	7.11	15.09	28.48	49.76	70.69	86.22	92.20	
	.01	.08	.35	.52	.40	.17	.10	
	.58	2.22	6.02	7.04	3.72	1.35	.80	
	7.60	17.51	19.49	15.15	8.69	.22	.00	
	84.10	64.50	45.06	26.93	15.90	11.44	6.30	
	.50	.50	.50	.50	.50	.50	.50	
	.10	.10	.10	.10	.10	.10	.10	
Total free and combined glycerine								
	13.39	13.21	12.41	9.54	5.53	2.24	1.24	
Calculated wt. of fatty phase per								
	100.00	100.59	101.01	99.80	97.32	95.22	94.70	
	13.39	13.29	12.54	6.25	5.38	2.14	1.17	
Calculated wt. of glycerine liberated to								
water phase per 100-lb, of coconut oil—lb	0.00	0.10	0.85	3.87	8.01	11.25	12.22	

TABLE V  $\sim$   $\sim$   $\sim$ 

of split is required, additional stages of boiling, settling, and water-phase removal are carried out. As noted earlier, the use f increased volumes of water shifts the equilibrium toward completion. Four stages of splitting are considered the economically acceptable maximum. For greater water economy a countercurrent usage of water is followed, the second boil water for example, being used for first boil, the third boil water being used for second boil, etc.

Stirton, Hammaker, Herb, and Roe (31) report on the comparative catalytic effect of 16 different laboratory-prepared reagents and list the descending order of activity as follows: alkylaryl sulfonates, sulfoxylyl fatty acids, petroleum sulfonates, and Twitchell reagents.

Gueniot (9) claims a high degree of hydrolysis, using phosphoric acid in place of sulfuric with less discoloration. He states further that the shorter chain fats and the unstaurated fats split most readily.

Mueller and Holt (28) conducted extensive studies on changes in the composition of the fatty phase during the Twitchell splitting of coconut oil. A summary of their results appears in Table V, and the progress of the reaction may easily be followed. Sample 1 was the original fat, 10 is after the addition of splitting agent, 20, 24, and 31 were taken during the first boil. and samples 41 and 45 after the second and third boil, respectively.

Hartman (11) reports that the reaction velocity rate approximately doubles for each  $10^{\circ}$ C, rise in temperature in the neighborhood of 100<sup>o</sup>C. Some of the reagents were deleteriously affected by temperatures of 150°C. (under pressure). Ueno *et al.*(34) report that unsaturated fats split more easily than saturated fats and that gums, proteins, and lecithin retard the reaction.

It is customary in handling low-grade fats to pretreat the stocks with sulfuric acid, settle, and draw off impurities prior to Twitchell splitting. Certain disadvantages are found in the Twitchell process: high usage of steam, darkening of stocks, introduction of unsaponifiables into the fat, long reaction times, production of glycerine waters of lower quality, and inability efficiently to split certain low-grade stocks.

### Batch Autoclave Process

The autoclave process, by utilizing steam under pressure, raises the reaction temperature and therefore markedly increases the reaction rate. This method is widely used throughout the world, although to a far greater degree outside the United States than domestically.

Vertical cylindrical steel reactors, heavily insulated,

20 to 40 ft. high, lined (or clad) with type 316 stainless steel or of solid stainless construction are charged with fat and 30–60% of its weight of water. While originally an alkaline catalyst such as  $ZnO$ ,  $MgO$ , or CaO was used, current practice tends to eliminate them. The batch is heated with an open sparge steam coil at from 150 to as high as 800 p.s.i.; the air is first vented to prevent oxidation and darkening of the fatty acids. Mechanical agitation may or may not be used to maintain a constantly changing oil-water interface for diffusion of glycerine and water. At the lower pressures either a mechanical agitator or a constant steam bleed is necessary to maintain a reasonable rate of reaction. At the higher pressures the importance of mechanical agitation is reduced because of the increased solubility of water in the fats and fatty acids but may nonetheless afford some advantages.

After being held at operating pressure for  $1\frac{1}{2}$  to 6 hrs., the batch may be  $88-90\%$  split and is blown through a flash tank to a settling tank where the glycerine water is drawn off. The fat is re-charged to the reactor with fresh water to obtain higher degrees of split, if desired, and the process is repeated. Countereurrent use of boil water again effects substantial savings. When operating in two stages at the lower pressures, 150-285 p.s.i., it is eustomary to settle the batch in the autoclave and effect a phase separation there, permitting the blowing off of sweet-waters without losing the heat content of the fat. At higher operating pressures this is not as practical because of the higher solubility of the water in the oil phase.

Since the autoclave process rarely uses more than two stages of boiling, as compared with two to four stages in twitchelling, the reversible reaction of esterification is more noticeable in the former where higher glycerine concentrations prevail. Water-washing of the split stock is commonly practiced to prevent reesterification and subsequent high residues upon distilling the split stock.

Hetzer  $(12)$  and Lascaray  $(18, 19)$  note the highly active catalytic effect of ZnO over that of any other basic catalysts. Walter (36) proposed a two-stage hydrolysis at 6-7 atmospheres, the first with regular splitting agent and the second with a basic catalyst. In those cases where alkaline catalyst is used, the split fat is treated with a mineral acid, usually sulfurie, to split the resultant soap and is then water-washed. Yonese  $(39)$  reports a more rapid splitting reaction with the shorter chain length fats. He notes too the accelerating effect on the reaction of iron contamination, but this phenomenon does not appear to have any practical value since the fats are badly discolored.

The batch autoclave process possesses the advantage

of much shorter reaction time and higher purity fatty acids and glycerine waters. This may be offset in some cases by the higher initial cost.

# **Continuous Concurrent Splitting**

The Eisenlohr process (8) provides for concurrent feeding of a mixed stream of fat and water through a coil maintained at 2,500-3,000 p.s.i. The split stock is then depressurized in a flash chamber. Approximately  $90\%$  split is obtained. It has no great applicability because of the high pressures and temperatures involved, the necessity of using multiple stages to obtain maximum split, and the low productive capacity involved.

# **Enzymatic Splitting**

The use of enzymes to split fats at low temperatures  $(90^{\circ}-95^{\circ}F)$  is not practiced to any great degree except on a few isolated oils. The fat is charged to an open lead-lined tank with 30-40% of water and usually 6% of an enzyme. For example, a fermented, ground castor bean and water mixture may be used. After adjustment of pH to 5 and addition of an activator, the batch is agitated for one to two days. The emulsion is broken with acid, and the fatty acids at 80-90% split are collected. Uniformity and reproducibility are poor, operating times are long, and the process is limited to fats which are fluid below the operating temperature.

### **Continuous Countercurrent Autoclave Process**

Steadily developed since the first Ittner (16) and **Mills** (25) patents appeared in 1938 and 1939, respectively, the continuous contercurrent high pressure process is the most efficient of the current methods of fat hydrolysis. Originally presented for operating temperatures of  $185^\circ$  to  $235^\circ$ C. in the presence of catalyst, the process has developed (4, 5, 6, 7, 13, 14, 15, 17, 22, 23, 26, 27, 29, 30) to its present high efficiency.

Two principles afford the process its advantages: high temperatures and pressures are used, giving short reaction time, and full countercurrent oil and water flow produces high degrees of split.

The equipment used for the process is the most costly of those listed. The heart of the system is a tower usually 20 to 48 in. in diameter and 60 to 80 ft. high, constructed originally of clad steels but more recently of solid type 316 or Inconel, built to withstand operating pressures of over 700 p.s.i., and well insulated.

The fat is introduced *via* a sparge ring at a point about 3 ft. from the bottom of the tower with a highpressure feed pump. The water used is introduced at a point similarly situated near the top of the column at a usual ratio of 40-50% of the weight of the fat (1, 2). The fat rises through the hot glycerine-water collecting section at the bottom of the column and passes through the oil-water interface into the continuous phase, the oil layer in which the hydrolysis takes place. Direct injection of high pressure steam is utilized quickly to raise the temperature to  $500^{\circ}$ F. Pressure is maintained at a level high enough to prevent vaporization of the water (700-725 p.s.i.).

The fat, as the continuous phase, rises in the column against a descending, dispersed stream of water. The water is usually dispersed in the fat in small droplets by means of a perforated plate near the top of the

column. The descending water is usually further heated with direct sparge steam in the upper section. The level of the interface is customarily maintained at a point usually 3-4 ft. above the oil inlet near the bottom of the column.

Pressure in the column is maintained by means of a back-pressure control valve in the fatty acid discharge line. The level of the interface is maintained by controlling the discharge of the water phase. The two phases after heat-exchange with the countercurrent feed at opposite ends of the column discharge to flash tanks, The fatty acid easily reaches 98% split and often 99% by virtue of the countercurrent contact of the most highly split fat with the water of the lowest glycerine concentration. Close control of the heat exchange at each end of the column is most essential for efficient separation because of the solubility of water in fatty acids and of the fatty acids in glycerine water at the extremely high operating temperatures.

The flashed split fatty acids normally contain no more than  $1\%$  moisture. The glycerine-water concentration usually rises from an average of about 24% before flashing to 30-32% after flashing. Feed rates of fat of 7,500 lbs. per hour are common. Mills (25) has presented an equation for calculating glycerine concentration *versus* water used in the continuous process.

$$
W = SY + (X) (54)/92 + X/C - X
$$

where

- $W =$  lbs. of water used per 100 lbs. of fat
- $S =$  lbs. of water-soluble in 100 lbs. of fat at the operating pressure
- $Y = a$  decimal representing the proportion of waterinsoluble fatty acids contained in the fat (This is approximately 0.95 for the tallow class and 0.90 for the coconut class.)
- $X =$ lbs. of glycerine liberated by hydrolysis of 100 lbs. of fat
- $C =$ lbs. of glycerine in 1 lb. of sweet water (usually 0.20 to 0.50)
- $54$  = molecular weight of moles of water
- $92 =$  molecular weight of glycerine

There are many variations of the basic process. Mills (23) combines splitting with distillation and recycles the residue back to the splitter. Leaders (20) combines splitting with fraetionation and crystallization.

The continuous countercurrent high pressure process splits fats and oils more effciently than other processes listed, producing high degrees of split, short reaction time (usually  $2-3$  hrs.), and little, if any, discoloration of the fatty acids. Because of the efficient internal heat exchange it affords very high steam economy. The large units can change stock completely in about 8 hrs. by stopping the flow of fat feed and increasing the water feed until all the fat is displaced. This however is impractical for short runs. Smaller size units now available  $(500 \text{ lbs.}/\text{hr.})$  remove the earlier objection of unsuitability for changing stocks.

The principal objection to this process lies in its high initial cost.

## **Methyl Ester Process**

Based upon the Bradshaw and Meuly patent (3). anhydrous methanol (approximately 60% excess over theoretical requirement), containing  $0.25\%$  to  $0.50\%$  alkaline catalyst (NaOH, KOH, NaOMe), is added to dry, neutral, or low acid oil at  $80^{\circ}$ 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**

JOHN W. McCUTCHEON, John W. McCutcheon Inc., New York, New York

THE PRINCIPLE of spray-drying soaps and detergents<br>is not new. In fact, the majority of installations<br>today do not differ greatly from those in use 30 is not new. In fact, the majority of installations today do not differ greatly from those in nse 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 (inoxpericnce regarding retailmarket requirements) or through a desire to avoid costly patent litigation.

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<br>a paste, and generally are **J. W. McCutcheon** 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 spraydrying tower is the vacuum type adapted for the production of toilet and other soaps where combined eooling 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 inchuding ducts, fans, inlet and outlet openings, and dust separators; heating system; slurry preparation and feed; product removal system; and controls, maintenance, and safety.

FEW GENERAL considerations on tower operation  ${\bf A} \;$  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 countereurrent tower. When the air flows in the reverse direction from top to bottom, the tower is known as concurrent tower.