High Temperature Processing of Fatty Oils and Acids*

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MANY of the processes for converting fatty oils into more valuable products require the use of high temperatures. They have much in common from chemical and engineering points of view, and it is interesting to consider them as a group. Certain things can be expected to happen chemically when fatty oils are subject to high temperature treatment and there are common plant design considerations. This discussion is directed to the consideration of fatty acids about C_{10} and higher and the corresponding glycerides. By "high temperature" we mean the range above the temperature which can be reached by the use of low pressure (100 pounds per square inch or less) steam. Some commercial applications of high temperature (hereafter referred to as HT) will be enumerated, and their similarities and differences will be considered.

Fatty oils of the drying type, such as linseed and tung, are treated to produce bodied oils, modified resins, and varnishes. Bodying of oil involves heating to an elevated temperature and holding at this level for the period of time required to increase the viscosity by polymerization to the desired point. This is usually done batchwise in agitated kettles, 1,000 to 2,000 gallons in size, heated by direct fire, electrical heat, or Dowtherm vapor, and cooled by water coils or recirculated Dowtherm. A typical unit for the production of bodied linseed oil is shown in Figure 1.

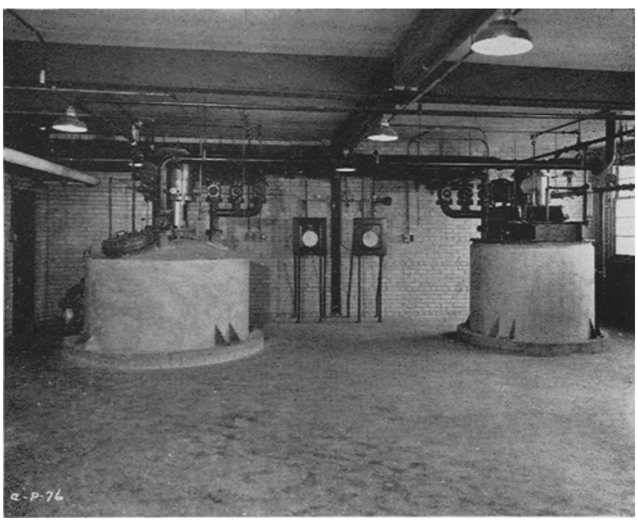

FIG. 1. Operating floor view of oil bodying kettle.

The oil bodying unit is at the left; the kettle at the right is for producing blown oil. The oil is heated as rapidly as possible; this is usually to save time, but for some products it is an essential part of correct processing. The oil is then held at a specific temperature, such as 575°F. until the desired viscosity is reached (as determined by tests made during operation), and then the batch is cooled quickly so that no further polymerization occurs. The curve in Figure 2 shows the heat-up time and cooling time which are typical for a 1,000-gallon Dowtherm heated and cooled oil bodying kettle. The holding time and top temperature can be set to suit the particular oil being handled and the degree of bodying desired. During the operation there are distilled off some water, the more volatile organic constituents, and some decomposition products. These are condensed and not returned to the kettle. The more of this material that can be eliminated, the better the quality of the oil will be with regard to color, odor, and acid number. Sparging inert gas through the oil during processing assists in carrying off the undesirable volatile material. Some companies carry out the bodying operation under vacuum to remove these fractions more completely.

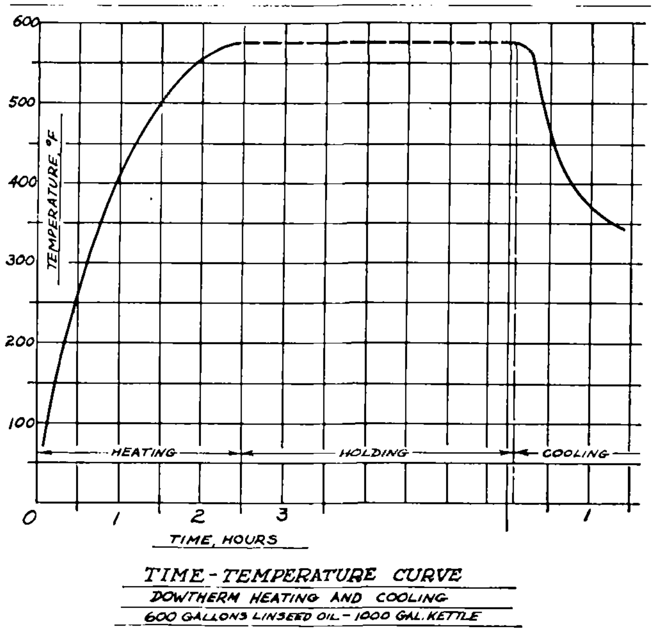

FIG. 2. Dowtherm heating and cooling curve for 1,000-gallon kettle.

When mixtures of glycerides, fatty acids, and alcohols are heated together at elevated temperatures, a group of reactions, known as interesterification, ester interchange, and alcoholysis, occurs. As an example, in the production of modified drying oils or alkyd resins various mixtures of drying oils, fatty acids, and glycerine are heated together at a temperature of about 400°F. for a period of time to effect the desirable chemical rearrangements.

SYNTHETIC resins of the oil modified alkyd type are made from: dibasic acids such as phthalic or maleic anhydride; polyhydric alcohols such as glycerine, glycol, and pentaerythritol; and drying oils such as linseed, tung, soybean, and dehydrated castor. The glycerine and oil are heated together to form monoglycerides, and then the phthalic anhydride is added. The cooking is carried on at about 500°F. to complete the esterification reaction (as indicated by removal of all the water of reaction) and give the desired viscosity. The water may be removed by heat alone or with the help of inert gas sparged through the batch. Another method now finding favor is azeotropic dehydration or solvent cooking. In this system the reaction is carried out with an entraining agent in the batch such as xylol which boils, carrying with

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it some of the water of reaction. The mixture is condensed, the solvent is separated from the water and is returned continuously to the kettle. After cooking is completed, the xylol remaining in the kettle is distilled over into a receiver. Figure 3 shows a typical

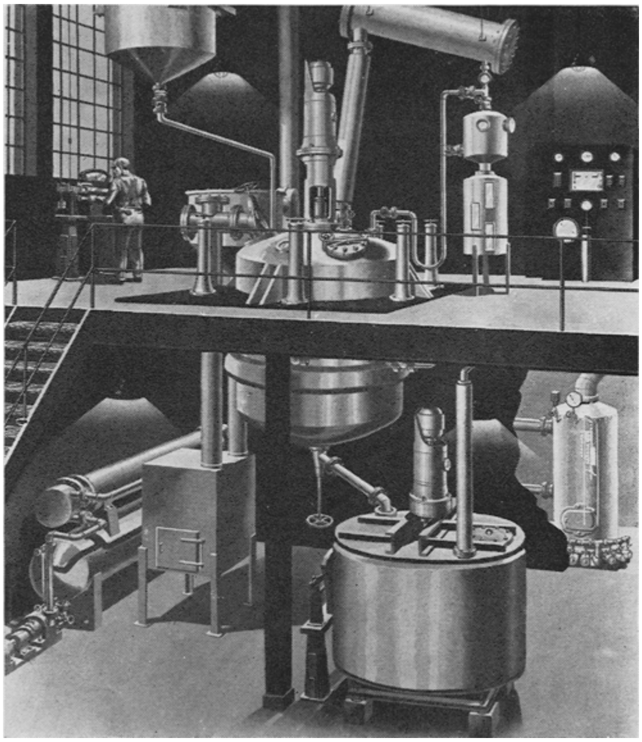

FIG. 3. Typical Dowtherm heated alkyd resin kettle with auxilliaries.

manufacturing unit for alkyd resins, including the agitated reaction vessel, Dowtherm heating and cooling systems, charging tank, run-down tank, solvent cooking system, and fume scrubber. With this set-up resins can be made either by fusion cooking or the solvent method.

Varnishes are mixtures of resins, either natural or synthetic, and drying oils cooked together. Their preparation formerly was conducted in small portable kettles over open fires but is now carried out in the same type closed kettles used for oil bodying and alkyd resin manufacture.

The possibility of bodying drying oils continuously has been discussed for some time, and a number of companies have carried out investigations along this line. No commercially successful technique is in operation yet although it seems quite possible that a suitable process could be developed, particularly if it were desired to manufacture large quantities of one grade of oil.

The splitting or hydrolysis of fats to fatty acids and glycerine is an important commercial process. The acids are marketed as such or are employed as intermediates for other products, such as soaps and fatty esters. Fatty acids are purified by distillation so higher grade products can be made from low grade fats by splitting and then distilling. Fatty acids are simpler chemical building blocks than the mixed glycerides which occur in nature; from the fats one can isolate the individual acids and then build up many chemical products. The Twitchell process, which was popular for many years, is now being replaced by continuous pressure splitting, which requires no catalyst and produces a high degree of split at lower operating cost.

In the Colgate-Emery process, shown in Figure 4, the fat and water are contacted countercurrently at HT and pressure in a stainless steel column. The tower is filled with fat, which is the continuous phase, the feed being admitted at the bottom. Water is fed in at the top in small droplets and, being heavier than fat, gradually falls to the bottom of the column. As the fat progresses upward, it is hydrolyzed and gives up its glycerine to the descending water. Fatty acid is discharged from the top, having first given up its heat in a heat transfer section to the incoming water. The sweet water is discharged from the bottom after first exchanging heat with the incoming fat. A typical layout of a fat splitting plant might be as shown on Figure 5. A recently constructed high capacity fat splitting plant is shown in Figures 6 and 7. One is an outside view with the splitting column at the left and the building containing the rest of the equipment at the right. The other is an inside view show-

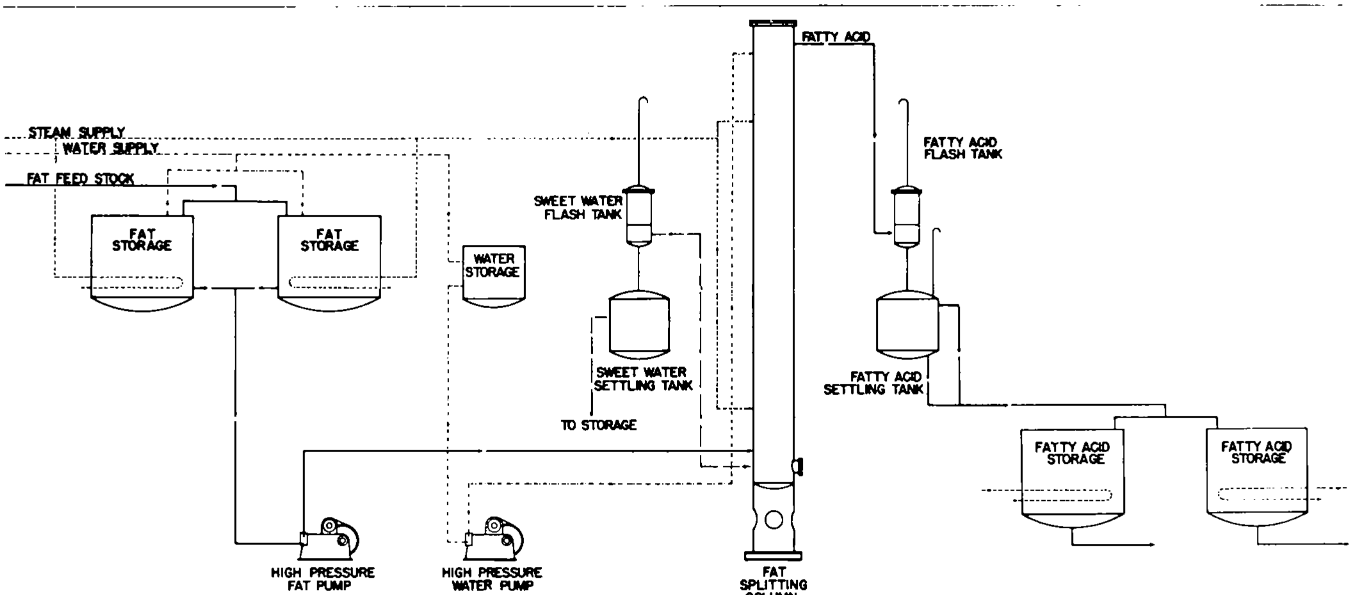

FIG. 4. Flow sheet—Colgate-Emery fat splitting process.

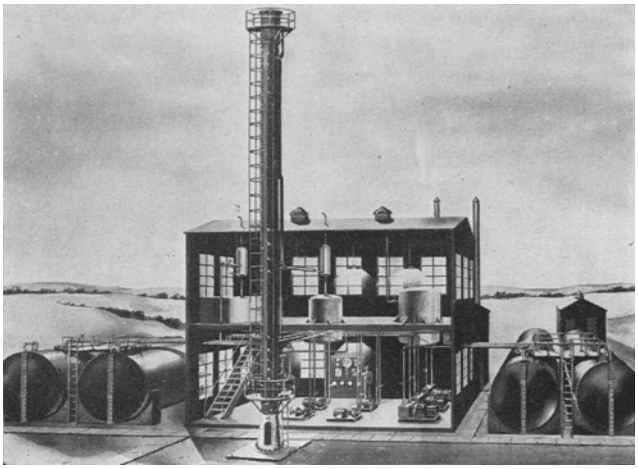

FIG. 5. Picture of typical fat splitting layout.

ing the transfer pumps and the high pressure feed pumps.

High pressure batch type fat splitting is suitable for relatively small scale operations. In this method approximately equal quantities of water and fat are heated with excellent agitation in a high pressure autoclave to a temperature of about 470°F. and a pressure of 500 pounds per square inch, under which conditions splitting occurs rapidly without catalyst. To obtain a high degree of split it is necessary to drain off the first sweet water and perform a second hydrolysis with another charge of water.

MOST edible oils and fats, and many industrial oils, are deodorized before being marketed. Deodorizing is a distillation process; the oil is heated to about 400°F., or higher, under an absolute pressure of about 5 mm. Hg and steam is sparged through the batch. When adequate odor removal has been achieved, the oil is cooled and discharged. Although the degree of removal of free fatty acids has been widely used as a measure of the extent of deodorization, it does not give a true picture and is not a substitute for actual odor and stability tests. A typical unit for the batch deodorization of oils is shown in Figure 8. Heating is either by Dowtherm vapor or by high pressure steam, and cooling is by liquid Dowtherm or water, either in the kettle or in an external cooler. A good arrangement is to have two sets of coils, one for steam heating and water cooling, and

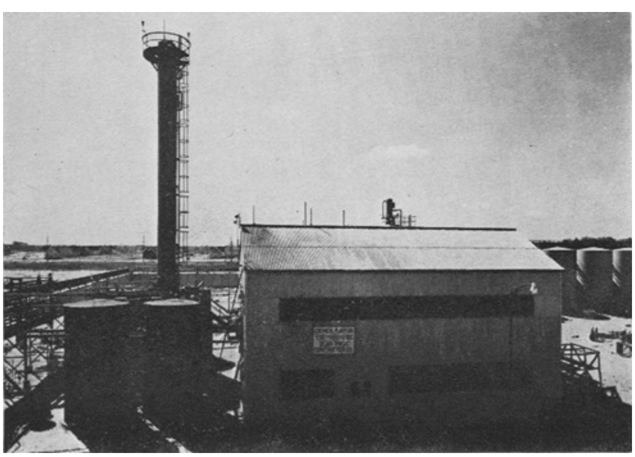

FIG. 6. Outside view of recently completed fat splitting plant.

one for Dowtherm heating. When using this system, both steam and Dowtherm are used until steam temperature is reached, and then the remainder of the heating is done by Dowtherm only. Certain oils have been successfully treated on a continuous basis which permits large production in relatively small equipment with good heat economy. Oils vary considerably in their response to deodorization treatment; for example, cottonseed oil is relatively easy but corn oil is difficult. The treatment of the oil before deodorization has an important effect. For certain oils a thorough filtration just prior to deodorization makes the treatment much more effective.

Fatty acids, after preparation by splitting, are usually purified by distillation. In simple distillation the mixed fatty acids are removed from the high boiling fraction containing most of the color and unsaponifiable material. In fractional distillation the mixed acids are separated according to molecular weight. Fatty acid distillation is usually carried out continuously, but batch-wise operation is also feasible.

A suitable unit for continuous simple distillation is shown in Figure 9. The fatty acid is dehydrated and

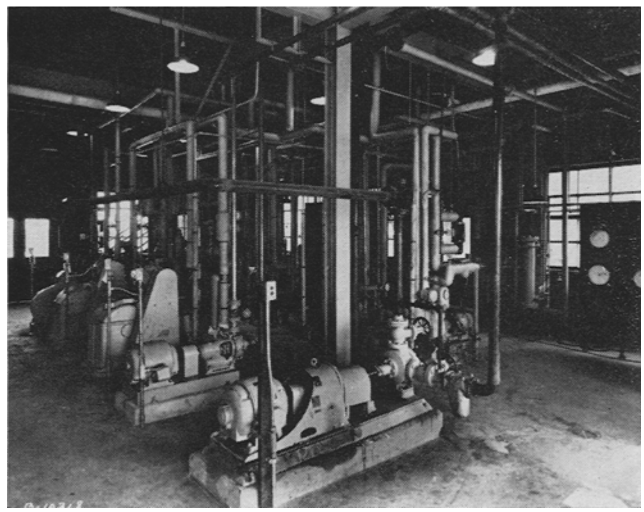

FIG. 7. Inside view of fat splitting plant showing pumps.

deaerated under vacuum, is pumped through a Dowtherm heated preheater, and then enters the top plate of a stainless steel stripping column. As the material drops down from plate to plate, it is heated by Dowtherm coils and its vaporization is aided by a stream of sparging steam which enters at the bottom of the column and passes upward. The fatty acid vapors are condensed while the water vapor and non-condensibles are carried through a vacuum system consisting of a booster, a barometric condenser, and a multi-stage vacuum jet. The fatty acid, as condensed, is sent to storage or to the next processing step. The residue leaving the bottom or the column is usually concentrated in a small batch-type still in order to recover some additional fatty acid and to produce a commercially saleable pitch. The entire distillation system operates under a high vacuum. When it is desired to deodorize the fatty acids at the same time, they are first passed through a small forecolumn where, at a lower temperature, and by the use of a small amount of steam, the light, odor-containing fractions are stripped off and separately condensed.

The separation of mixed fatty acids into fractions according to molecular weight can be done by the usual fractional distillation technique in bubble tray columns under high vacuum although the equipment must be constructed somewhat differently than that which is used for petroleum products. The hold-up in the column should be kept quite low so that the retention time is short and so that there will be little thermal decomposition which adversely affects the color and odor. A special case of fractional distillation is the purification of tall oil, which is obtained as a by-product in the manufacture of paper from pine wood by the Kraft or sulphate method. Tall oil is essentially a mixture of fatty acids (mainly oleic, linoleic, and linolenic) and rosin acids (mainly abietic). Tall oil, after a sulphuric acid treatment, is fractionally distilled under high vacuum, employing sparge steam to assist in the separation. The fatty acids are condensed from the vapors leaving the top of the column, and the rosin acids are recovered at the bottom.

IN general, all distillation processes for fatty materials are carried out at high vacuum or with a sparge of steam or inert gas, or both. Vacuum and steam sparge are to a certain extent interchangeable. If a certain distillation process can be carried out satisfactorily at 5 mm. absolute pressure without a sparge, the same effect might be produced at 10 or 15 mm. with a stream of sparging steam. Inert gas sparging is ordinarily used only at about atmospheric pressure, but it gives the same effect as some degree of vacuum.

Fatty acids react with alcohols, both mono- and polyhydric, to form useful esters. They are used in perfumes, flavoring materials, plasticizers, resins, emulsifiers, and wetting agents. By splitting fats, isolating the individual fatty acids, and then re-esterifying with glycerine, tailor-made fats are obtained. Many of these are better than natural fats for specific purposes. Esterification of fatty acids with various alcohols, glycols, and glycerine is ordinarily handled in batch fashion in a jacketed agitated kettle heated by Dowtherm or some other indirect means. The condensing system is designed to return alcohol to the kettle but remove the water of reaction. The finished product is cooled in the kettle or can be discharged hot through a separate cooler. Usually the reaction is carried out with an excess of alcohol, which is removed by vacuum distillation after the reaction has been completed.

Although the common hydrogenation operation of "hardening" fatty oils is carried out at relatively low temperature, some hydrogenation jobs require more drastic treatment and high temperatures are used. If it is desired completely to saturate a fat or fatty acid mixture, a HT can be employed for rapid reaction. A suitable apparatus for the batch-wise hydrogenation of unsaturated fatty acids would consist of a Dowtherm heated stainless steel autoclave, designed for 200 or 300 pounds per square inch operating pressure.

Fatty alcohols are prepared by the hydrogenolysis of fatty materials at high pressure and temperature. Fatty acids, glycerides, or esters are treated with hydrogen in a batch or a continuous autoclave at about 3,000 to 4,000 pounds per square inch pressure and about 600°F. temperature in the presence of suitable catalyst. Fatty acids are converted to alcohol and water; glycerides to alcohol and glycerine; and esters to a mixture of both alcohols.

It is of interest to mention a few special types of

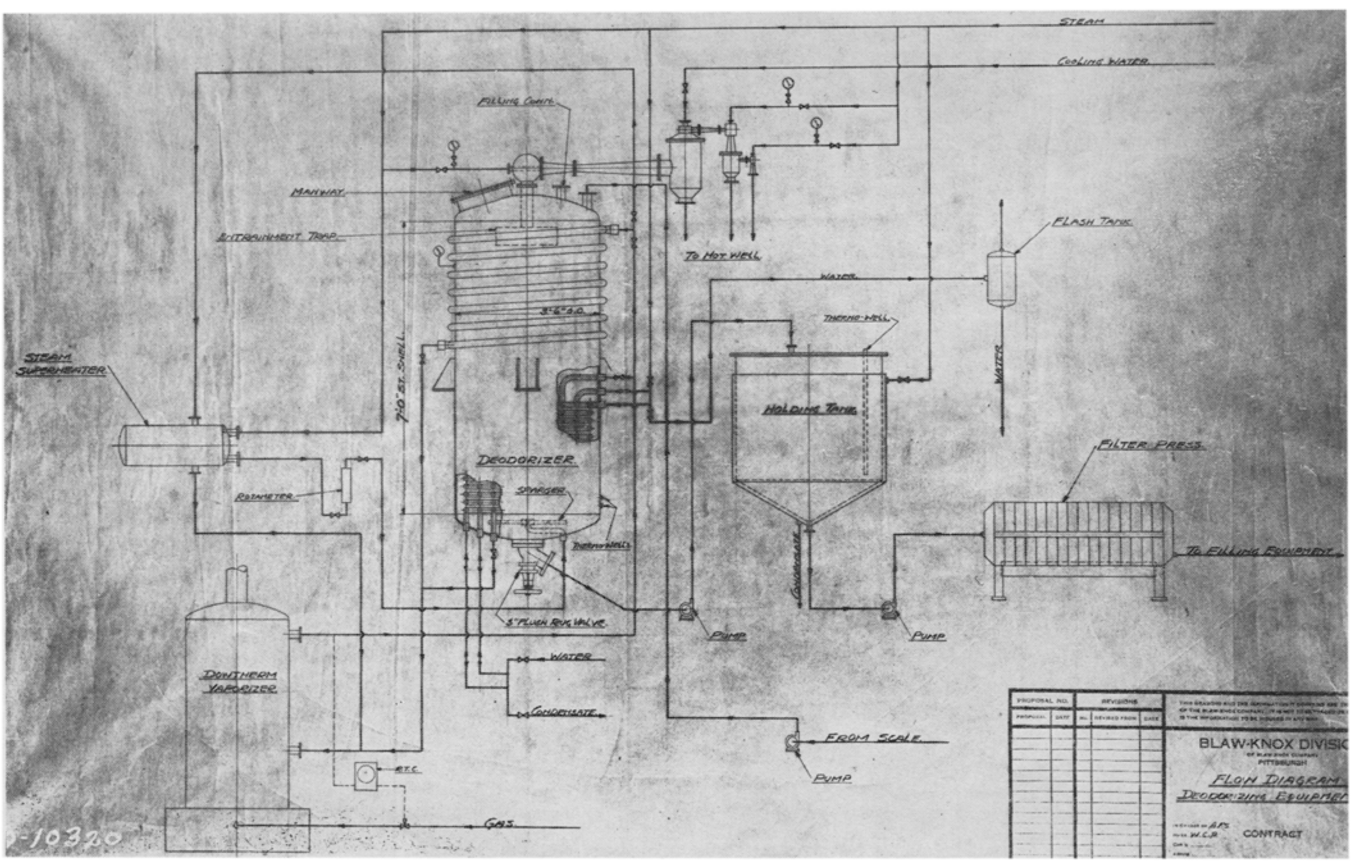

FIG. 8. Flow sheet of batch deodorizing system.

processing without describing them in detail. Oiticica oil, in the crude form, is a solid material; a usable drying oil is produced by heating it for a period of time at an elevated temperature which causes a chemical change, liquefying the material. Castor oil is dehydrated by heating it at an elevated temperature with catalyst to produce a drying oil having very desirable properties. HT greases are prepared by forming soaps of certain metals such as lithium and then incorporating at HT suitable lubricating oils. Many of these special greases are produced in Dowtherm heated or direct fired equipment as contrasted to steam heated vessels used for the usual type of grease.

Methods of heating which may be used for HT fat processing include direct fire, electric heat, high pressure steam, Dowtherm circulated as a liquid or vapor and various heat transfer liquids, such as hot oil, high pressure water, fused salt, and sodium-potassium alloys. The primary sources of heat are ordinarily gas, oil, or electricity. The selection of a heating system should be based on a careful analysis of all factors for the particular process and locality under consideration. One should consider cost of fuel, first cost of heating system, maintenance expense, ease of control, ease of starting and stopping, and flexibility for varying process conditions. The most popular heating systems are direct fire, Dowtherm, and high pressure steam. Each has its advantages and disadvantages, and all three should be considered when planning a new operation.

DIRECT fire frequently has the advantage of simplicity and low first cost. Possible disadvantages include fire hazard, non-uniformity of heating, and poor temperature control. The use of vigorous agitation in direct fired kettles improves the operation in many respects, and this part of the design should receive careful attention. Operations requiring the use of volatile solvents or other hazardous materials, for example, the cooking of alkyd resins by the azeotropic dehydration method, are not well suited to heating by direct fire. It is not good engineering to have a direct fired unit in the same room where volatile explosive materials are being handled. Electric heat (with strip type or radiant heaters) often permits a neat self-contained installation with good temperature control and fairly good uniformity of heat application. The energy cost is high, and there is some fire hazard.

High pressure steam has the general advantages of an indirect medium—good temperature control, heating uniformity, and absence of fire hazard. The high pressure needed for the temperature range being considered puts a serious limitation on its use. Where water enters into the reaction, as in fat splitting, it is an ideal heating method. Dowtherm has the same advantages as steam with the added feature that HT can be reached with very moderate pressure. Its use has solved many heating problems. Any indirect heating system has a built-in safety factor; the temperature cannot go above that in the heating coil or jacket. Even if agitation or liquid flow should stop, there is little possibility of a dangerous condition occurring. With indirect systems it is frequently possible to heat small batches in large kettles as the application of heat right at the liquid line will not cause decomposition since the temperature difference is small. If the materials being processed are particularly heat sensitive, the jacket can be divided

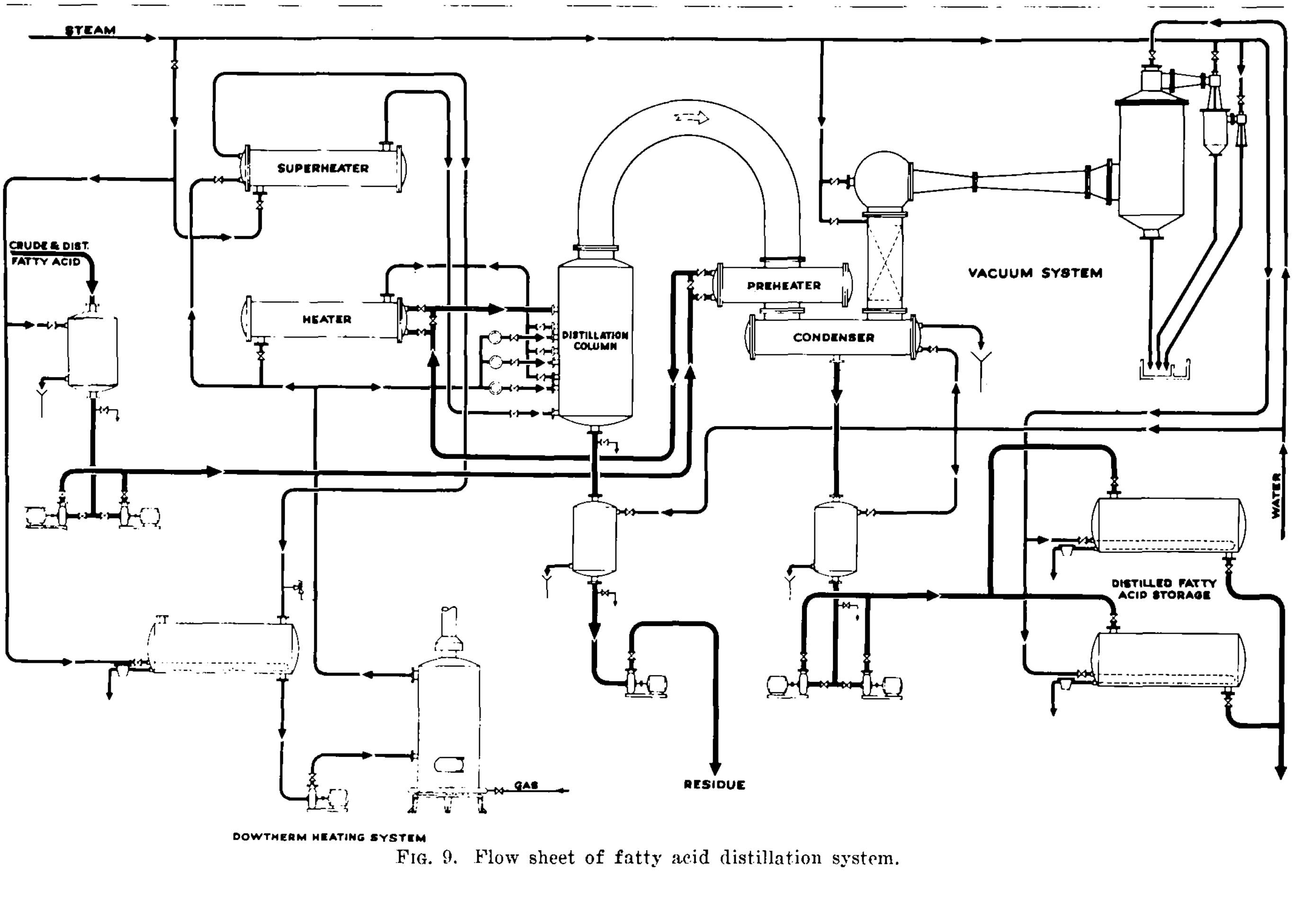

FIG. 9. Flow sheet of fatty acid distillation system.

into two compartments to permit heating at different levels.

A type of high temperature heating that is designated by the term "Electro-Vapor" is shown in Figure 10. In this system the jacket of the heat user is made somewhat larger than usual and contains a bath of Dowtherm which is boiled by electric immersion heaters. Electrical energy input to the heaters is controlled by the temperature of the batch and the pressure in the jacket. The unit is a combination of a heat user and a vaporizer built into one. A photograph of a completed kettle of this type is shown in Figure 11. The Electro-Vapor system combines the advantages of electric and Dowtherm heat. It is a simple self-controlled unit with no external piping or boilers and gives the heating uniformity characteristic of Dowtherm. The unit is explosion-proof and can be mounted in a small space anywhere that can be reached by electric wiring. The ease of control is excellent, and it is readily adaptable to full automatic operation.

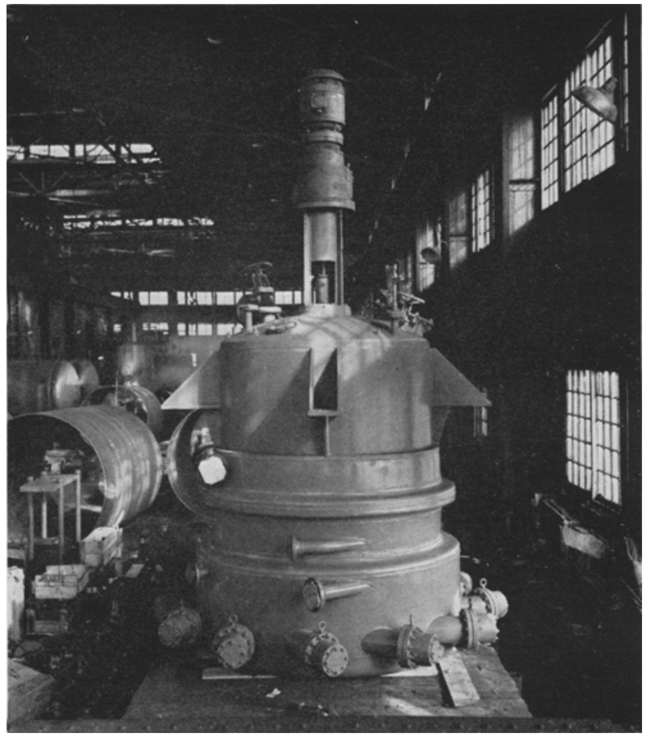

FIG. 11. Photograph of electro-vapor heated kettle.

The proper control of HT is of prime importance to successful processing, and the problems are greater than in low temperature systems. It is common practice to use controllers actuated by thermocouples rather than the usual type of liquid or gas filled thermometer systems. Valves used to control the flow of HT heating fluids require considerable maintenance in order to keep them in top operating condition; they tend to stick and leak at the stuffing boxes. Many HT processes are controlled manually for the reason that suitable automatic controls are quite expensive. Temperature controls and safety devices for limiting pressure must be of the most reliable types because the consequences of failure are extremely serious. Special precautions must be taken to prevent operation of high temperature equipment in the wrong sequence. Safety devices are of considerable assistance, but in general the workman must be properly instructed. For example, if a wrong valve were opened admitting

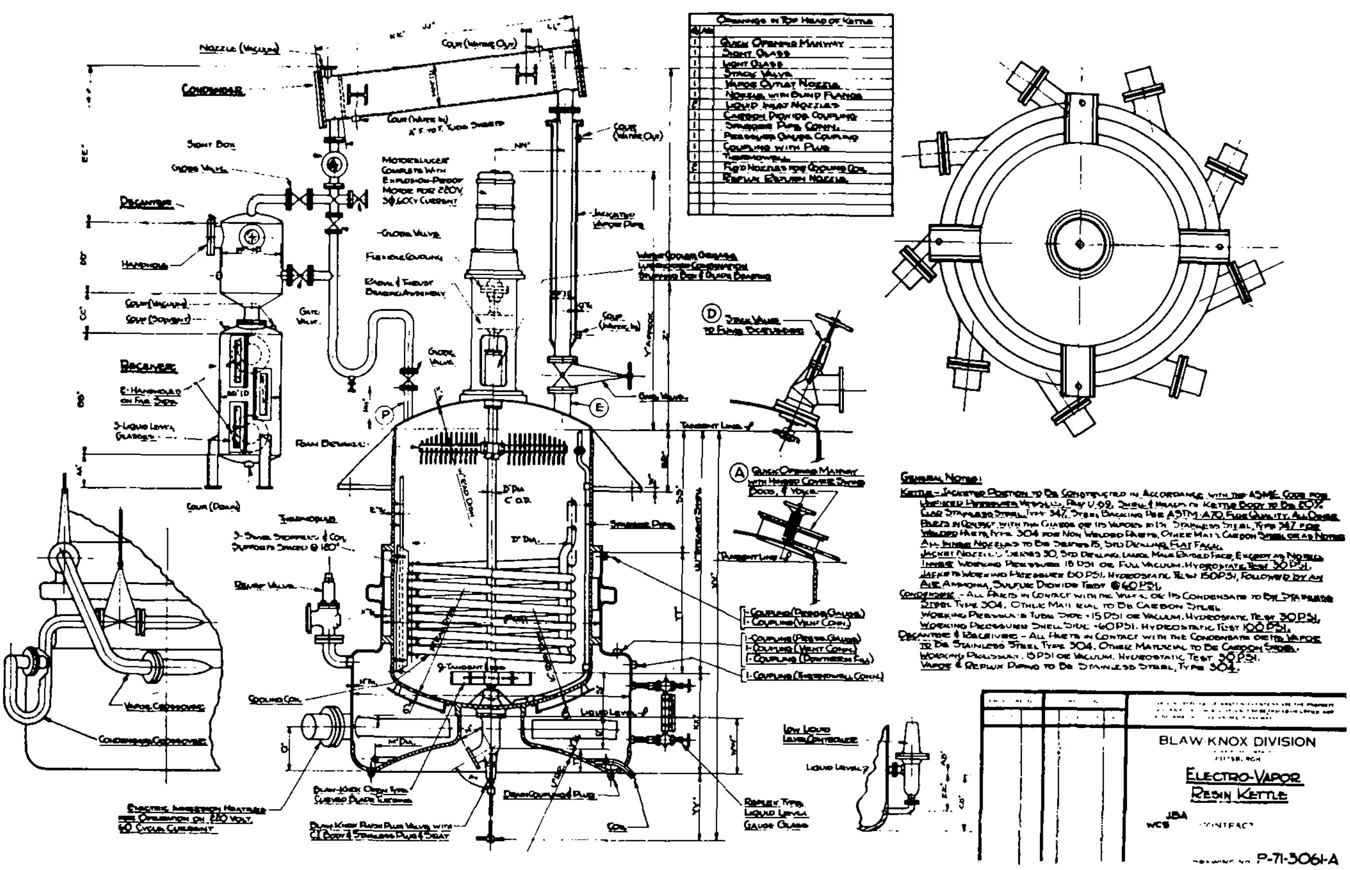

FIG. 10. Drawing of electro-vapor heated kettle.

a quantity of water to a batch of oil at 600°F., the effect could be disastrous.

The product of a HT reaction must be cooled before storage or packaging or further processing unless the subsequent treatment also requires HT. In a continuous process this is done by heat exchange possibly followed by water cooling. A batch process requires high capacity cooling which operates only at intervals; it must take a HT material down to a low temperature rather quickly to save time and to check the reaction. This may be done with an internal coil or an external jacket, using recirculated Dowtherm or sometimes water for the cooling medium. Even though it is not necessary for process reasons to cool the hot material discharged from a kettle, it may be necessary to cool the kettle or ventilate it in order to open the manway and charge the next batch without fumes filling the room.

TO elevate materials to HT requires a large amount of heat. Also most products must be cooled before exposure to air or before packaging. By the use of heat exchange a large proportion of the thermal energy is saved, and at the same time all or most of the cooling can be accomplished. The control of heating and cooling is facilitated since control is applied to only a part of the load. Many HT processes would be uneconomical without efficient heat exchange, and others can be made more profitable by the use of it. Heat exchange is especially applicable to continuous processing; there are few cases where heat exchange between raw material and product can be used to advantage in batch operation. The most common variety of exchanger, and the most efficient for many purposes, is the shell-and-tube type. The tube side is usually made multi-pass to give high liquid velocity; the shell side is provided with baffles for the same purpose. Differential thermal expansion can be provided for by a floating tube sheet or bell-type expansion joint in the shell. If either the shell or tube side is subject to fouling, coking, or corrosion, access openings or complete disassembly should be provided for inspection, cleaning, or tube replacement.

When the materials being exchanged are immiscible, the contact can be direct instead of through tube walls. This has been employed to advantage in the Colgate-Emery fat splitting process. Heat transfer by direct contact is quick and efficient and can readily be built into the reaction vessel if desired.

Thick and efficient thermal insulation is a characteristic feature of all HT processing units. This is necessary for the conservation of heat, keeping the building at a suitable temperature, and protection of personnel. Thicker insulation is used on continuous units than on batch apparatus that is alternately heated and cooled.

Some of the reactions of fatty oils, such as polymerization, hydrogenation, and oxidation are exothermic, and the heat generated must be continuously removed to control the temperature. Others, as in the case of fat splitting, are either endothermic or involve little heat of reaction. In such cases heat must be added to keep the temperature at the right level.

Fatty oils and acids are relatively inert at low temperatures. Elevated temperatures are required for rapid reaction with water, oxygen, hydrogen, and alcohols and for polymerization. Also the vapor pressures are low at ordinary temperatures so that the processes involving evaporation or distillation require HT, vacuum, and sparging steam. The reactions of a fatty material at high temperature may be beneficial and tend toward the desired product or may be detrimental and produce undesired by-products. Except for a few specific cases where oxidation is desired, as in the production of certain drying oil products, oxygen and its detrimental effects are carefully excluded. In general, oxygen causes undesirable reactions resulting in increased color, bad odor, off-taste, or instability. Before subjecting oils to HT, they are often deaerated by warming and then subjecting to a vacuum. High grade products are stored under a blanket of inert gas. The leakage of air at agitator and pump stuffing boxes and valve glands should be minimized.

Many high temperature fatty oil processes produce as a by-product obnoxious fumes which can seriously affect the health and attitude of the workmen and cause complaints from residents in the community and municipal authorities. Adequate fume control equipment should be employed using condensation or scrubbing for vapors and electrostatic precipitators, incineration, or impact scrubbing for smokes.

In all HT processing the time-temperature relationship is very important. A batch of a certain fat can be hydrolyzed to the desired degree in two hours at 500°F.; at 475°F. a much longer time is required and at 525°F. it can be done more quickly. In general, for the HT processing of fatty oils there is not a single exact temperature for the reaction, but an optimum must be selected, based on considerations of quality, equipment cost, and heat economy. Frequently there are two factors to be considered, one of which is favored by temperature and the other adversely affected. In the hydrolysis of a drying oil, such as linseed, HT increases the rate of splitting but also increases the rate of polymerization which is undesirable. Optimum conditions must be selected to give a suitable hydrolysis rate without undue

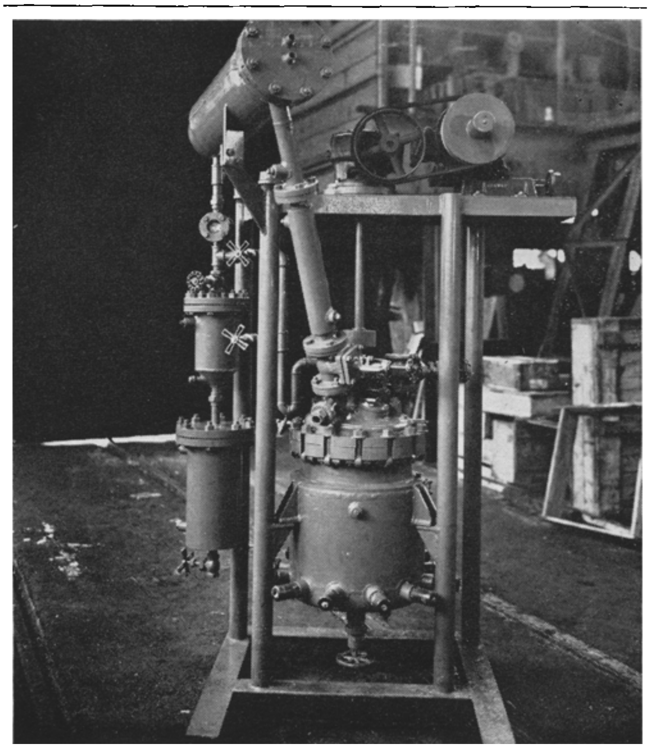

FIG. 12. Pilot kettle for high temperature reactions.

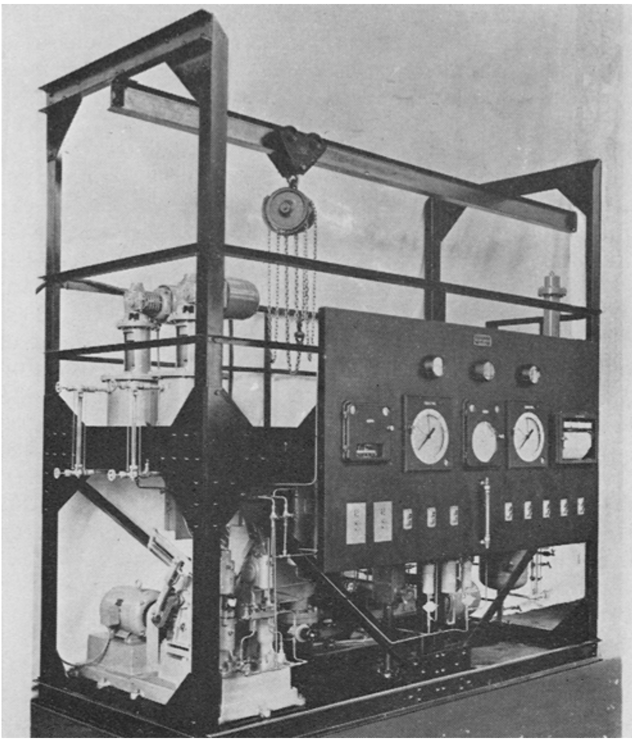

FIG. 13. Pilot plant for continuous high temperature high pressure processing.

polymerization. In the deodorization of oils a HT increases the rate at which the undesirable materials are removed but also increase the rate at which odorous materials are formed due to thermal decomposition. In continuous deodorization the decomposition is minimized by reducing the retention time.

Equipment using fatty oils as raw materials must be made flexible so that it can handle quite a variety of materials. Frequently the particular oil used depends upon the market condition; most of the fatty oils are interchangeable to a certain extent. For example, salad oil might be corn oil, soybean oil, or cotton seed oil, depending upon the price. Stearic acid might be manufactured from animal oils or hydrogenated vegetable oils, depending upon price.

ONE of the most difficult and important decisions in planning a new process is whether it should be batch or continuous. In general, a continuous system is for large scale production for one particular product; and a batch system is suited for smaller production capacities and has greater flexibility.

The following points should be kept in mind when deciding whether to employ batch or continuous equipment for a particular processing operation:

For Batch

1. Equipment costs less for small scale operation.
2. Frequently easier to control end point.
3. Equipment can usually be used for other purposes if desired.
4. Easier to start and stop.
5. Can often be operated to advantage on one or two shifts, instead of three.
6. Can make one or two batches and then shut down.
7. Laboratory to plant development takes less time and is less expensive.
8. Formulation or processing procedure can be changed quickly.
9. Shut down for repairs or maintenance is easier and less expensive.
10. Sometimes less blending is required for standardization.
11. Minor process modifications to correct for differences in raw materials are easier.
12. Individual batches can be kept separate if desired.
13. Equipment is frequently less complicated and less liable to get out of order.
14. Does not require reworking of material produced when starting or stopping production campaign as in case of continuous operation.

For Continuous

1. Heat economy is good because of efficient heat exchange.
2. For large production the equipment cost is low per unit of output.
3. Requires less space.
4. Requires less labor.
5. Because of countercurrent operation a greater reaction efficiency or greater product concentration is possible.
6. Can be operated readily out of contact with air or moisture.
7. Since no fast heat up is required, the peak loads for heating are less and the heat generation equipment is smaller. This is especially important in the case of electrical heating.
8. Less storage is frequently required.
9. Less inventory of material in process.
10. Retention time can be smaller, which sometimes improves quality.
11. Some processes are possible only on continuous basis.
12. Waste disposal sometimes easier with continuous small flow.
13. Less liability for error due to human element.
14. Product is frequently more uniform than is possible by batch methods.
15. Often the product is of higher grade than is possible by batch operation.

Batch type equipment can frequently be designed so that it can be used for more than one process. For example, it is reasonable to build a high temperature autoclave system in such a way that it can be used for fat splitting, fatty acid distillation, hydrogenation, and esterification. If you wish to build a plant involving several high temperature processing steps

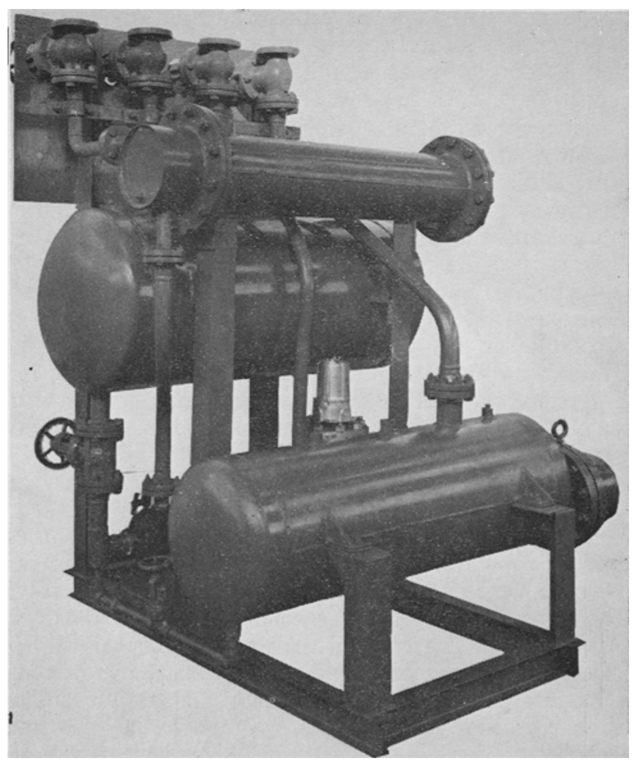

FIG. 14. Dowtherm heating and cooling system for pilot use.

but wish to start off on a small scale and increase it later, it can sometimes be done as follows: Build a single flexible piece of equipment to handle all of the operations, one at a time. Then as additional production is required put in separate units for the individual steps, still employing the original item for one of these. In this way no equipment is discarded, and a logical step-wise program can be employed.

Some processes are operated to advantage on a semi-continuous basis which means that one of the reacting materials passes through continuously while the other is retained in the equipment for a specified length of time.

It is frequently difficult to decide whether to use a single large unit or a number of small ones to turn out a certain production. Listed below are some of the points that should be considered.

Several Small Units

1. Can start out on small scale and then increase production later by adding additional units.
2. Less pilot work required.
3. Can turn out small orders economically.
4. Opportunity of staggering to minimize peak loads for steam, water, electricity, water disposal, etc.
5. Less danger in case of fire or explosion.
6. Can still operate even if one unit is out of commission.
7. Frequently better control.
8. Quicker heat up and cooling.

One Large Unit

1. Total equipment for a certain production costs less and requires less space.
2. Less operating labor.
3. Less testing and standardizing per unit of production.
4. Less metallic contamination.

THE proper selection of materials for HT fat or fatty acid processing is of prime importance. For fatty acids at HT, stainless steel, especially Type 316, and Inconel, are suitable; for low temperatures, aluminum is satisfactory. For neutral glycerides, carbon steel can frequently be used. Copper is now considered a poor material of construction because of its effect on the product; edible products, for example, are very sensitive to copper, which affects the taste, odor, color, and stability.

The material of construction must be suitable from the standpoint of corrosion resistance as well as the effect on the material being processed. For cases where one has not had previous experience, it is best to rely upon the results of corrosion or contamination tests made under operating conditions. Many equipment manufacturers are glad to furnish samples from commercial batches of available material containing welds to simulate the material that will be found in an actual fabricated vessel. If possible, select a material of construction which is available commercially and which is accepted by regulatory codes as a suitable and reliable material. Practically all conditions which you will encounter can be satisfied by one or more of the following materials:

a. Carbon Steel.
b. Aluminum.
c. 18-8 Stainless (Type 304 for non-welded vessels and Type 347 for welded items).*
d. Type 316 Stainless.*
e. Inconel.*

The use of techniques already developed for various kinds of HT fatty oil processing will in some cases make it possible to work out new processes in this field without employing pilot plant tests. One must be cautious, however, about departing too far from present practices, and pilot work certainly has its place in handling reactions which are vastly different from anything that has been done before. Since many of the HT processes are similar, pilot

* For certain purposes these materials can be used as integral cladding on a carbon steel backing.

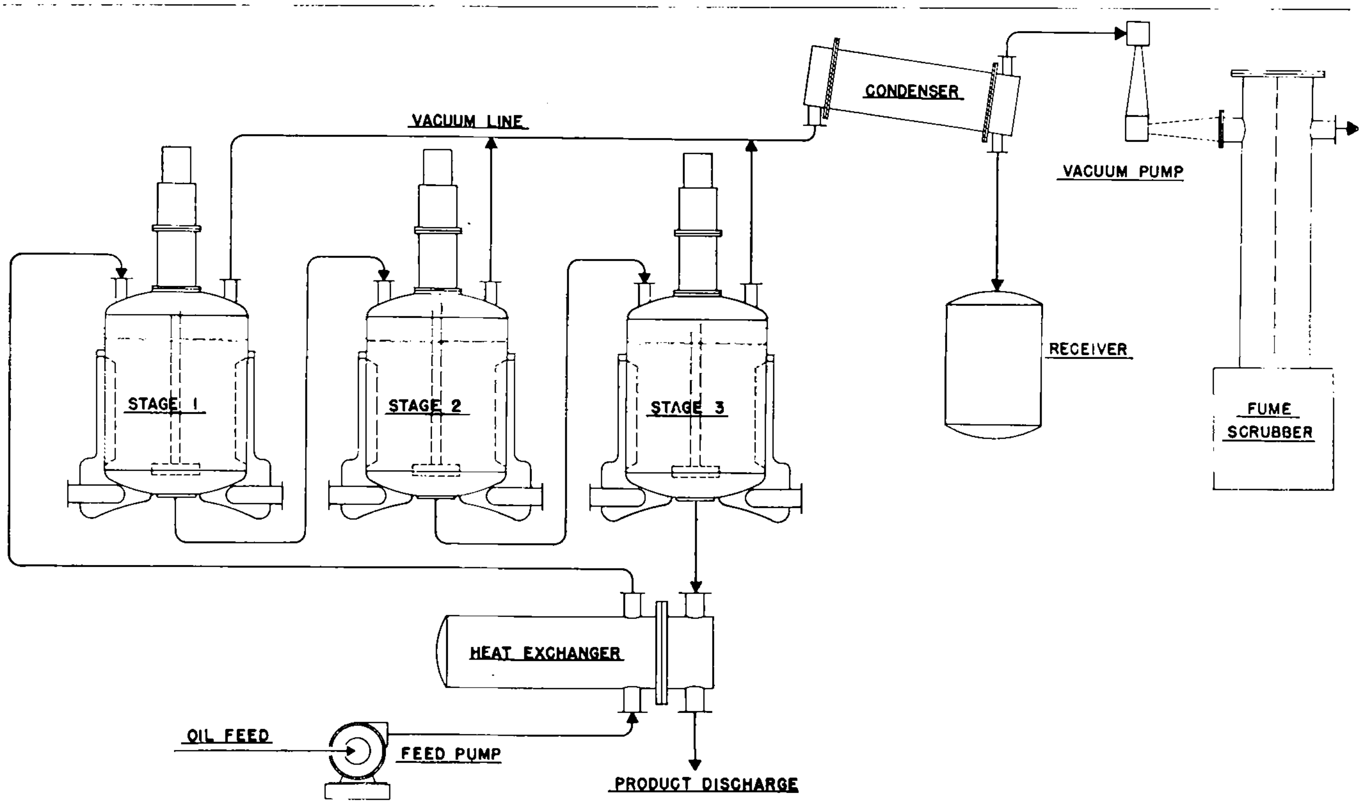

FIG. 15. Continuous oil bodying system.

units can frequently be made quite versatile and be employed for a variety of studies. The batch type unit shown in Figure 12 was designed especially for oil bodying, alkyd resin making, and varnish cooking, but it can be used successfully for almost any type of high temperature work within the pressure limits of the unit, such as esterification, hydrogenation, deodorization, and fatty acid distillation. Figure 13 shows a high temperature and high pressure continuous reaction unit consisting of charging tanks, feed pump, heat exchanger, multi-compartment agitated autoclave, blow-down tank, and instruments. This particular unit is for temperatures to 650°F. and pressures to 4,000 pounds per square inch.

Figure 14 shows a standard self-contained Dowtherm heating and cooling system built especially for pilot work. This is portable, and it is only necessary to connect the heated equipment to the four valves.

It is frequently desirable to have a convenient pilot plant unit available even after the commercial plant is placed in successful operation since its use will permit improvements to be made or can guide the way to changes which are necessitated by using a different raw material or different product specification.

When oils are heated to high temperature at rapid rates, the oil film next to the heating surface is at a temperature considerably higher than the main body of the oil. This film tends to carbonize and build up in thickness until it reaches a point where it seriously interferes with heat transfer; then the surface must be cleaned. For example, fatty acids in a distillation process may be heated in a tubular exchanger with direct fire or Dowtherm. At the heat transfer rates ordinarily employed the film next to the tube wall is enough hotter than the main body of the fatty acids to cause gradual carbonization or coking. If the flow is interrupted for even a few seconds, the condition is aggravated. Coking should be minimized by proper equipment design and operation, and the heaters must be constructed so that any coke which accumulates can be readily removed.

Many of the products under consideration are either solid or quite viscous at room temperature. A common feature, therefore, of fat processing equipment is that many of the product lines and receivers are jacketed with steam or hot water so that the material is maintained fluid. Condensers are frequently cooled by liquid Dowtherm or hot water so that solidification will not occur in the tubes.

HT processing of fatty materials constitutes a possible hazard to personnel and property. Fatty oils are inflammable, and the apparatus and storage tanks contain quantities sufficient to cause serious fires. Fatty materials at the higher temperatures will burn immediately upon contact with air. A leak in the equipment, or a burst pipe can produce a fire without the assistance of a separate igniting source. If air is suddenly admitted to a vessel by opening a manway, or by collapse due to vacuum, a fire can occur. If water is suddenly introduced into a quantity of hot oil, the evolution of steam can be so violent as to constitute an explosion.

Rupture discs or safety valves should be mounted directly on the equipment being protected and the discharge should be piped to some point where a sudden flow of hot material could not cause danger to personnel or could not be ignited by a flame. Pipes to and from safety devices should be heated, or periodically inspected, or handled in some way that they cannot become clogged with solid material, thereby rendering the safety device inoperative.

Process equipment used for high temperature and high pressure service must be of quality higher than that used for ordinary operating conditions. The service is much more severe and the consequences of failure more serious. It is poor economy to use anything but very best equipment for these conditions.

As pointed out, the various HT processes for treating fatty oils have certain things in common and a study of the various individual members of the family should serve as a valuable guide in solving new problems in this field. A typical problem will show what can be done with some of the general rules.

AS previously stated, the continuous bodying or polymerization of drying oils is not yet considered to be successful on a commercial basis. Now, based on what one has learned in studying other processes, what might such a system look like? It requires heating to a certain temperature, say 600°F., holding the oil at this temperature for a period of time, depending upon the particular oil being processed, and the degree of body desired, say one hour, and then cooling rapidly to stop the reaction. The heating and holding should be done under such conditions that dissolved and entrained air and water are removed and low boiling fatty acids and decomposition products are released. This is best done under vacuum. For the continuous process to be successful there should be little or no back-mixing of the material; in other words, each particle of oil should be retained for approximately the nominal holding time. Heat exchange and automatic instrument control should be employed to effect heat economy and minimize the operating cost. A flow sheet of a possible unit for this purpose is shown in Figure 15. It consists mainly of a series of three small agitated kettles heated by Dowtherm. The incoming oil is raised to about 500°F. by heat exchange with the product and is admitted to Stage 1 where the heating to 600°F. is completed rapidly and is retained for one-third of the total holding time. It then passes successively to Stage 2 and then 3. The reason for the separate stages is to prevent back-mixing. Each kettle is thoroughly agitated and operated under vacuum so that volatile materials are released and removed. The vapors are condensed and this liquid, which is rich in certain fatty acids, can be sold or used for other processing. The discharge from the vacuum system goes to a suitable fume scrubber which removes any smoke carried by the non-condensible gases. The product leaving the third stage is cooled by heat exchange with the feed oil. As indicated, the kettles are heated by the Electro-Vapor method since with a continuous unit and the use of good heat exchange, the electrical heating load is not great and the use of this type of system permits a very neat self-contained layout. Also, the separate vessels can be operated at slightly different temperatures if it is desirable from a process point of view. Would the continuous oil bodying system work? The writer believes that it would.

The same general line of reasoning can be applied to many HT fatty oil processing problems. By employing techniques that have been developed for similar operations, one can design a plant unit for a new process with considerable confidence.