Fractional Distillation

R. BERGER and W. McPHERSON, EMI Corp.,
3166 Des Plaines Ave., Des Plaines, IL 60018

ABSTRACT

While practically all the fatty acids produced in the fatty acid industry are distilled products, these materials are all, at least to some degree, fractionated fatty acids. Rarely indeed are today's fatty acids suited for any of the many applications to which they are put without the quality and homolog distribution improvements which only fractional distillation can guarantee. Thus, this separation is of vital importance within the fatty acid and derivative industries. Fractional distillation is industrially a practical separative method for: (a) 16:0 and 18:0 fatty acids, such as those derived from hydrogenated fats and oils like tallow, soybean, cottonseed soapstocks, palm oil and others; (b) 18:0, 20:0, 22:0, and 24:0 fatty acids from hydrogenated fish oils or high erucic rapeseed oil; and (c) 8:0, 10:0, 12:0, and 14:0 fatty acids from the hydrogenated fatty acids from the lauric oils group (coconut, palm kernel, babassu, etc.). While theoretically possible under idealized conditions in the laboratory, it is not practical to separate palmitic, oleic, heptadecanoic, and stearic acids by means of fractional distillation

Fatty acids have been distilled to purify them for over a hundred years. A simple distillation or fractionation removes odor and low boiling unsaponifiable matter (low boilers); triglycerides, polymerized products, color bodies, hydrocarbons and other breakdown products (high boilers). The low boilers pass through the product condenser, the high boilers remain in the still as pitch. Early stills without the aid of vacuum illustrate the property of fatty acids that make any fractional distillation difficult–heat sensitivity. Temperatures higher than 250 to 270 C, along with time, decompose fatty acids into anhydrides, ketones and hydrocarbons, or polymerize unsaturated fatty acids to dimers, trimers, etc.

In the 30s, high vacuum steam ejectors (with a range of 1–6 millimeters of mercury) became available for commercial installations, which simplified straight distillation in that temperatures under 250 C could be maintained in the still without using great quantities of injection steam.

Simple batch distillation (Fig. 1) used today for small capacity plants has many disadvantages, one being the length of time fatty acids are subjected to the high temperatures of the heating surface. The most obvious fault is that without fractional distillation or purifying trays, some high boilers will get over to the product condenser. This type of unit distills the fatty acids from an ever increasing concentration of foots or residue. Deaerated feedstock is automatically fed in as distillation progresses. When the color of the product condensing darkens, because residue is being distilled, feed to the unit is stopped and the temperature of the still increased to recover as much fatty acids as possible from the residue. Product from the condenser is kept separate during this time to be run again through the still. High losses and poor quality of distillate as compared with continuous distillation stills are common disadvantages of direct fired or radiant-heated pot stills.

Continuous straight distillation (Fig. 2) will produce a satisfactory product if odor or low boiling compounds are not present to any large extent. This unit continuously distills fatty acids under automatic controls. Deaerated feedstock is preheated and enters the distillation column and is quickly vaporized by flowing across heated trays aided by steam rising up through the column. Vapors are purified of high boilers by two stages of entrainment elimination and by washing with recycled distillate. The product is condensed on low pressure drop packing by recycled product automatically cooled to a temperature that allows low boilers and steam to pass to the light end condenser. The light end condenser works the same way as the product condenser with the temperature set to allow steam to pass through without condensing.

High boiling compounds along with the bottoms from the distillation section pass through additional heated trays and a reboiler to strip them of fatty acids. This type of unit will give a good quality distilled fatty acid but does not attempt

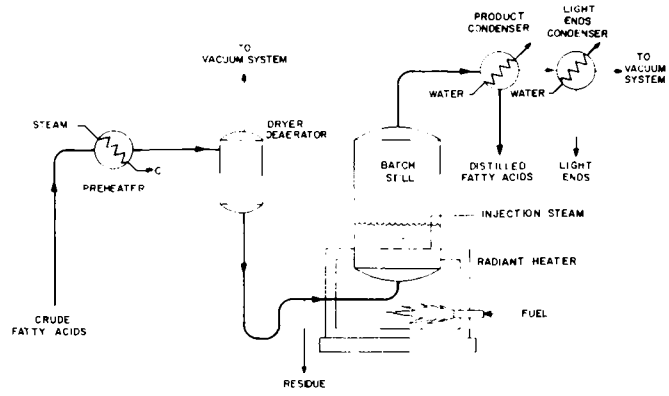

FIG. 1. Simple batch distillation.

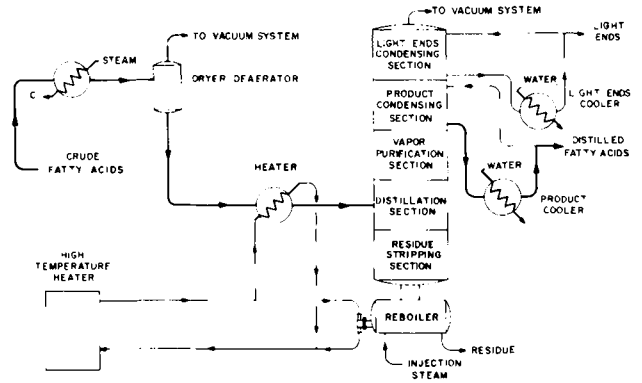

FIG. 2. Continuous straight distillation.

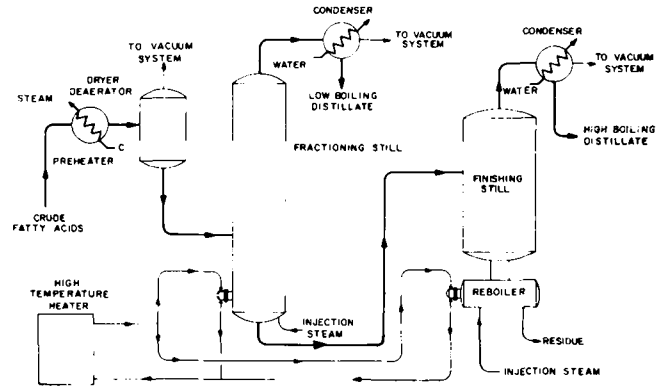

FIG. 3. Continuous fractional distillation.

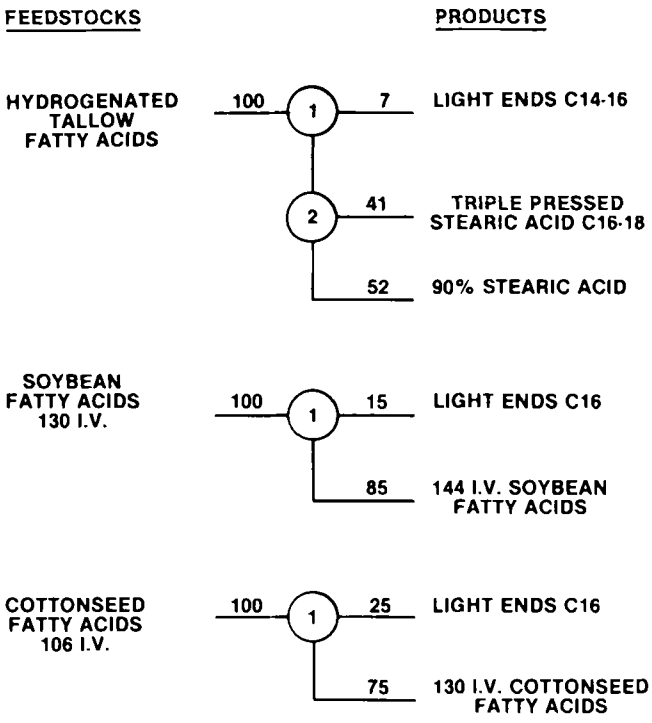

FIG. 4. Partial fractionations.

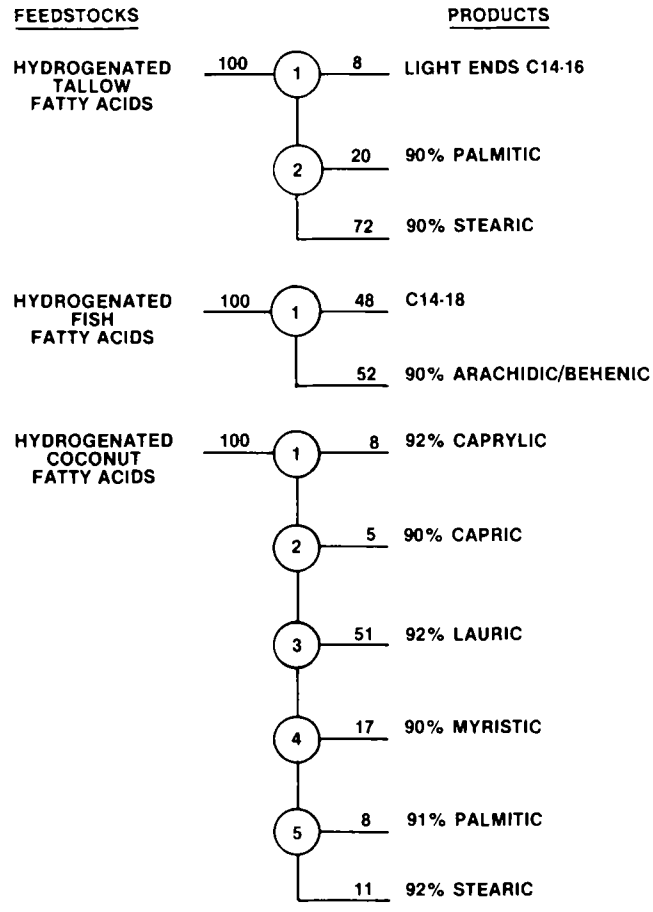

FIG. 5. Full fractionations.

to fractionate fatty acids. Distilled fatty acids, however, can be put through this unit a second time, and by adjusting reflux ratios and temperatures separate high and low boiling fractions can be obtained with the low boilers collected in the product section and the high boilers brought out the residue section.

Because all fractionating trays have a pressure drop of from 1–2 mm, and fatty acids are heat sensitive, fractionating stills are limited to ca. 20–30 trays, depending on the vapor pressure of the fatty acids to be separated. Any more trays would cause pressure in the reboiler section to be higher than 30–50 mm. The vaporization temperature at higher absolute pressures may exceed the decomposition temperatures of the fatty acids. Because of this limitation to get high purity fatty acids from two to six fractionating stills may have to be used for continuous operation.

Figure 3 shows a fractionation system for crude or distilled fatty acid feedstocks. The low boiling fraction is condensed off the top of the first still, and the high boiling fraction from the bottom of the fractionation still is sent to a second stripping still. The high boiling product is distilled and condensed off the top of the second still.

Nonvolatile impurities originating in the crude feedstock or traces of nickel catalyst from hydrogenated feedstocks, along with polymerized materials formed in the fractionating still, are taken out the bottom as residue.

Fractionation stills are almost always custom designed to suit available feedstocks and product requirements. With lauric type fatty acids from coconut or palm kernel oils, up to 30 trays can be used for highest purity fractions because of the higher volatility and greater stability of the short chain saturated acids. Long chain fatty acids from fish oils and high erucic content rapeseed oil have much lower vapor pressures and would need low pressure drop packing or a limited number of fractionating trays to keep the reboiler below the decomposition temperature.

Molecular stills or thin film stills are used for simple distillation of heat sensitive products of low volatility but cannot perform clean separations that require more than one equilibrium stage.

Figures 4 and 5 show commercial products that can be obtained from fractional distillation stills.

Interrelationships in Fatty Acid Processing

E. FRITZ, Process Development, Union Camp Chemical Div., PO Box 570, Savannah, GA 31402

ABSTRACT

Examples are presented of the effect of each processing step on the quality, yield and throughput in the fatty acid processing scheme. These examples serve to illustrate the importance of seemingly unimportant process variables upon the economics involved in fatty acid processing.

Fatty acids are seldom sold or used as a crude product. They are more frequently processed through a series of unit operations and processes. These are depicted in Figure 1. As in most series of processes, the operation of each has an effect upon subsequent ones.

Most of our raw materials are byproducts of other industries and as such, are generally variable in quality. For