The Removal of Stearin From Fatty Oils With Liquefied Hydrocarbon Gases

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Introduction

MOST liquid fats and vegetable oils contain varying amounts of stearin, palmitin and other solid fats which interfere with their use for many purposes. Thus cottonseed oil contains about 16-18% stearin and palmitin which, if allowed to remain in oil used for edible purposes, will separate as a mush when the oil is stored in a cool place. With salad oils, this problem has become more acute in recent years since the advent of domestice iceless refrigerators with their lower temperatures. The separation of stearin from beef fat in making oleo oil is another familiar operation. The manufacture of inedible lard oil requires the removal of most of the stearin and other high melting fats to make it suitable for many industrial uses in such products as blended lubricating oils which must have a low pour point, and in lamp oils which must remain fluid at fairly low temperatures.

The methods commonly employed for removing stearin involve pressing or draining the oil from a solidified body of fat at the desired temperature. The principal drawbacks to the method are the low rate of cooling required in order to obtain large stearin crystals from which the oil can be separated, and the difficulty of getting effective separation of the oil, which is usually fairly viscous at the temperature of separation requiring careful control of pressures and low filter rates. The use of light diluents to reduce the viscosity of the oil and increase the rate of separation has apparently never been practiced. It is true that solvents have been used in extracting oils from seeds, etc., but not in the separation of stearin, probably because their use introduces the problem of diluent recovery and in the case of edible oils, the problem of flavor injury due to residual traces of the diluent or to the high temperature necessary to completely remove the diluent from the oil. Then too, there is the cost of the diluent to be considered which is largely a function of the efficiency of its recovery.

To overcome these disadvantages in the use of diluents, we have investigated the liquefied hydrocarbon gases, particularly propane and butane. These substances being unreactive chemically and substantially odorless do not injure the oil for edible purposes. Because of the low boiling point of the liquefied gases, they can be completely removed from the oil at relatively low temperature. Their cost is very low, thereby making diluent investment and replacement costs reasonable. They enable the obtainment of close temperature control with almost any desired degree of self-refrigeration by merely controlling the pressure and thus regulating the evaporation of the diluent. In addition, they appear to have an anti-solvent effect on the stearin, thus increasing the sharpness of the separation obtained. They are naturally handled in a vapor tight system which prevents the access of air, thus precluding any possibility of rancidity occurring during the destearinizing operation. The work which we are reporting here is of a preliminary nature only and was done to determine whether it is possible to completely crystallize stearin and palmitin in solution in fatty oil, consisting chiefly of olein, with the aid of liquefied hydrocarbon gases without causing the separation of appreciable quantities of olein. We have found that this can be done quite readily by properly controlling the temperature within a fairly narrow range as the following results will show.

Experimental

Destearinizing Cottonseed Oil—

The cottonseed oil used in this work had been given the usual caustic wash, and was a stock of high haze test such as commonly filtered on plate and frame presses for the removal of the margarine.

The low boiling point hydrocarbons most readily obtainable and applicable to the problem of separating the hard from the soft fats, are n-butane, i-butane and propane. Table I shows their boiling points, specific gravity and vapor pressures at ordinary temperatures.

TABLE I

Boiling Points, Specific Gravities and Vapor Pressures of n-Butane, i-Butane and Propane

	B. P.° F.— 0 Lb. G.	Density at 60° F.	Vapor Pressure at 70° F.
n-Butane	32°F.	0.585	17 lbs. sq. in.
i-Butane	10°F.	0.565	31 lbs. sq. in.
Propane	—44°F.	0.515	109 lbs. sq. in.

The diluents used in the work on cottonseed oil were the normal and iso-butanes. These were chosen because at temperatures below +10° F. either of them can be handled experimentally in open vessels. Preliminary experiments showed that the temperature for the separation of the fats should be approximately —10° F., with a concentration of 70-75% of diluent by volume.

The separation may be effected either by filtration or settling but since our preliminary work indicated that the handling of filter cakes might prove troublesome on a large scale, the major part of the work was confined to separation by settling.

The experimental procedure followed was to put the cottonseed oil in solution, cool, and allow to stand until the solution above the layer of settled solids became clear. The clear solution was then drawn off, the diluent removed by distillation; and the diluent free oil tested for haze at 32° F. The results are shown in Table II.

TABLE II

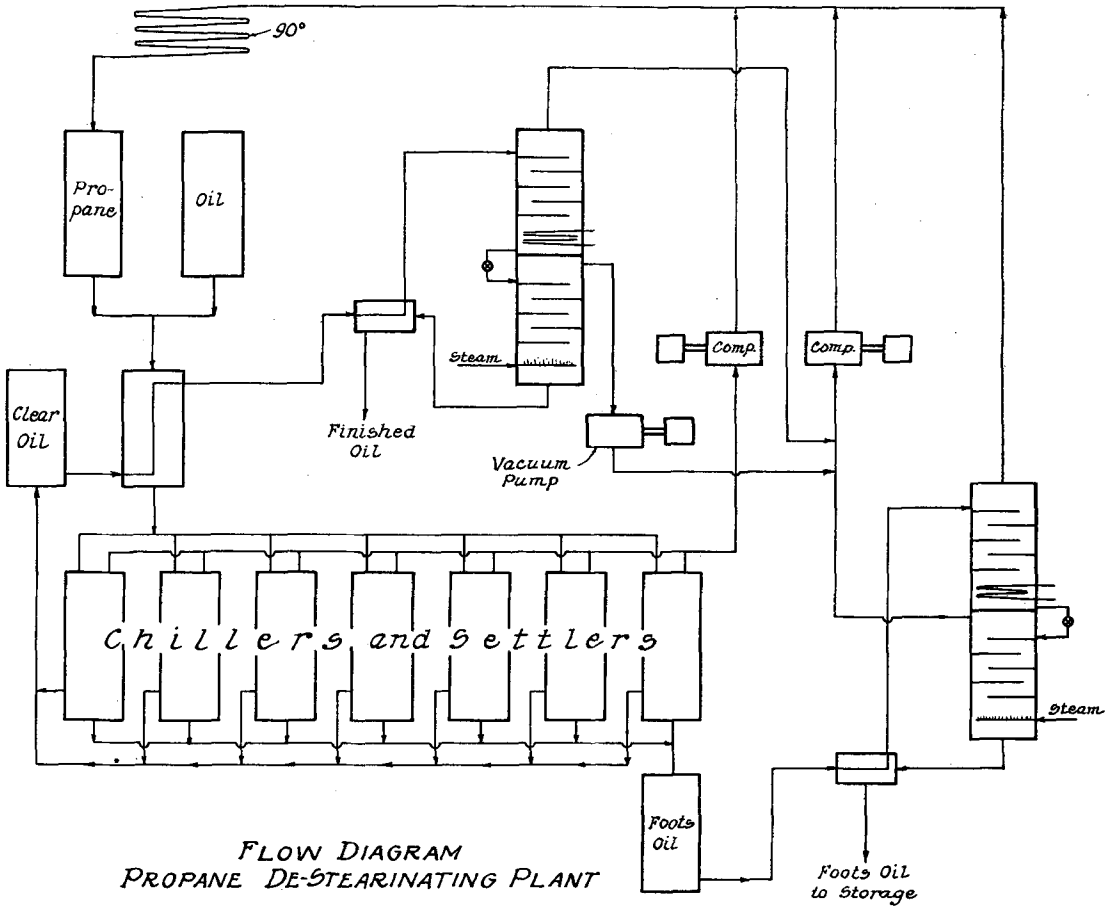
Settling Cottonseed Oil in Normal and Iso-Butane Solutions

% Diluent	Settling Temp. ° F.	Yield of Clear Oil, %	Clear Oil—20 Hrs. at 32°F.
75% i-Butane....	—10	65	No haze
70% n-Butane....	— 6	48	No haze

The Separation of Stearin from Animal Fats—

With animal oils such as beef fat the temperature of separation of oleo oil from stearin was found to be much higher than with cottonseed oil. With diluent concentrations of 70 to 75% by volume a separation into approximately 50% stearin and 50% "oleo oil" is obtained at approximately 30° F. Settling is exceedingly rapid, settling a distance of 10 inches in ten minutes.

A sample of beef fat was dissolved in propane in a closed vessel and settled at 30° F., the propane concentration being 72% by volume. A sample of the clear



FLOW DIAGRAM
PROPANE DE-STEARINATING PLANT

solution was removed, the propane evaporated, and the titre point of the fatty acids determined after saponification. The titre point was found to be 36.4° C.

The titre point of the "soft oil" could no doubt be varied by changing the temperature and concentration of the solution settled.

The method proposed for large scale operations with the animal oils is quite similar to that described for destearinizing cottonseed oil. For edible oils it seems that propane would be a more satisfactory solvent than the butanes because it would be easier to remove at relatively low temperatures. In considering this process we have assumed that the edible oils should be heated no higher than 130° F., and in the diagram of the process, shown in Figure I, we have added a vacuum steam stripping unit to the diluent recovery units to insure complete removal of propane.

According to this design, propane and oil in the proper proportions are pumped from storage through a mixing device, and the resulting solution precooled by exchange with cold clear solution. After leaving the exchanger the solution passes into one of the tanks which serve both as chillers and settlers. When the chiller is filled, the compressor, which is designed to operate under a variable suction pressure, is connected to it. The vapors are pumped off, condensed in a water condenser, and run back to storage. When the proper temperature is reached in the chiller the compressor is cut off and the chilled solution allowed to settle. When settling is complete the clear solution is pumped out through a side draw to the clear oil tank from which it is fed to the vapor recovery tower. After exchanging with fresh solution, and with hot finished oil, the stream enters the upper section of the tower where it is heated with closed steam. The propane flashes off under about 200 lbs. per sq. in. pressure, the temperature of the vapors being about 110° F. The oil and a small amount of propane passes into the lower section at 250° F. and is flashed at atmospheric pressure and stripped with live steam at about 215° to complete the removal of the diluent. The gas is picked up by a compressor, compressed to 200 lbs. per sq. in. pressure and recondensed.

The settled solid material is pumped out of the settlers at the bottom and treated in an identical manner to recover the diluent.

In order to be able to operate the distillation equipment and the compressors continuously a multiplicity of chilling and settling tanks are shown. By that scheme one tank is being filled, another is being chilled and still another is being emptied at all times.

An estimate of the requirements necessary to produce 90,000 lbs. of destearinized cottonseed oil per day is given in Table III. The requirements for destearinizing beef fat or a similar product would of course be similar.

TABLE III

Requirements for a Plant Producing 90,000 Lbs. of Destearinized Cottonseed Oil Per Day

Basis —Settling at —10° F. in 75% propane solution to yield 65% of destearinized oil.
Charge—138,500 lbs. per day.
Labor —1 operator and 1 helper per shift.
Power —Compressors, 80 h.p.; pumps, 25 h.p.
Steam —45,000 lbs.
Propane make up (assuming 0.5% loss)—350 gals. per day.

While this investigation has been preliminary in nature, the results indicate very strongly that in the case of cottonseed oil higher yields of cloud-free oil can be obtained than with the present destearinizing methods. Also lower cloud points than are now possible should be readily attainable. Beef fat and similar stocks can be separated into hard and soft fractions with a sharper separation than is now obtained and oleo oils should be obtainable by the use of propane with lower titre points than are obtainable by the present method. The process is adapted to the processing of large quantities of material with a significant reduction in labor requirements.

In conclusion we wish to acknowledge the kind assistance of Dr. R. C. Newton who supplied us with the fats and made helpful suggestions.

On inquiry whether all of the oil was removed from the stearin, Mr. Voorhees replied that some of the liquid oil was retained in the stearin. No second washings were made.

Mr. Cluff inquired if the titer was obtained on the stearin.

Mr. Voorhees replied no. He commented that the stearin cake left after evaporation of the hydrocarbons was somewhat watery and hard to handle. It could be filtered but the operation would have to be carried out in a closed system.

Mr. H. C. Dormitzer asked if any cloud tests for haze had been made on the destearinized oil other than the 20-hours at 32° F. test. Also, how much the yield of soft oil would be increased if the testing operation was carried out at a temperature which would just enable the oil to pass the test of 5 hrs. at 32°.

Mr. Voorhees replied that no tests have been made other than the 20-hours at 32°. He expressed the opinion that the yield could be increased by washing the oil and mixing it with fresh oil for further treatment. At the same time a drier stearin would be obtained.

Over 20,000 Displays Entered In Palmolive Soap Contest

Displays Doubled Sales, Say Many Dealers—$12,000 in Cash Prizes Awarded.

More than 20,000 dealers submitted photographs of displays in the Palmolive Soap Display Contest which closed November 30 and in which judging has just been completed.

Of these, 1,616 were selected to receive the $12,000 cash prizes. The remaining contestants will all receive a Palmolive Gift Box. Every state and virtually every locality in the country was represented in the competition.

The contest, the first of its kind sponsored by Colgate-Palmolive-Peet Company, was praised by officials of the company, by the judges, and by contestants as a great stimulus to the sale of Palmolive Soap.

E. H. Little, Vice President in charge of sales and advertising of the company, in announcing the prize winners, said:

"Although it has been impossible, thus far, to make a complete analysis of the results, a survey made among 178 of the contestants showed that their displays doubled their sales of Palmolive. We have received statements from many dealers saying that during the time their displays were up, their sales of Palmolive soap increased as much as five or six times.

"Practically every display had some unusual feature about it. The contest surely has created interest in better displays. It has focused the attenton of the dealer on the fact that good displays increase sales, not only in toilet soap but also on all classes of merchandise—such as coffee, canned goods, mayonnaise, cereals and other items.

"We are highly satisfied with the results and we hope we have brought the sales value of good, mass displays dramatically to the attention of the trade."

The contest, which called for the use of Palmolive Soap only in the making of a display, lasted for three months. To equalize the opportunities of the contestants, stores were placed in one of four groups, according to the size of the city or town in which they were located, and a complete set of prizes, from $250 for first, down to 300 of $5 each, were offered for each group.

Judges were Louis Sommer, prominent Omaha grocer; Gerritt Vander Hooning, a director of the National Association of Retail Grocers and also prominent in the retail business in Grand Rapids, Michigan; and John C. Sheehan, President of the Minneapolis Retail Grocers' Association. They were chosen for the job by Charles H. Janssen, Secretary-Manager of the National Association of Retail Grocers.

Their task, they admitted, was one of the hardest of their careers. Mr. Vonder Hooning said, "Never in my life have I seen so many unusual displays."

The judges were brought together in Chicago during December for the final selection of winners.

Production of Linseed Oil During the Quarter Ended December 31, 1933

The Bureau of the Census announces there were 24 mills in the United States which crushed flaxseed during the quarter ending December 31, 1933, reporting a crush of 189,266 tons of flaxseed and a production of 133,905,936 pounds of linseed oil. These figures compare with 139,934 tons of seed crushed and 90,987,258 pounds of oil produced for the corresponding quarter in 1932; 199,149 tons of seed and 130,478,580 pounds of oil in 1931; 206,944 tons of seed and 131,256,804 pounds of oil in 1930; 278,525 pounds of seed and 182,227,710 pounds of oil in 1929; and 313,346 tons of seed and 206,273,130 pounds of oil in 1928.

Stocks of flaxseed at the mills on December 31, 1933, amounted to 75,171 tons, compared with 87,384 tons for the same date in 1932, with 104,192 tons in 1931, with 125,218 tons in 1930, with 121,782 tons in 1929, and with 214,578 tons in 1928. Stocks of linseed oil reported by the crushers were 119,656,272 pounds on December 31, 1933, compared with 90,409,811 pounds for the same date in 1932, with 123,626,578 pounds in 1931, with 83,035,584 pounds in 1930, with 99,738,526 pounds in 1929, and with 120,724,853 pounds in 1928.

"Manufacture of chewing gum has been simplified through use of air conditioning. Vaults where postage stamps are stored have been air conditioned to eliminate loss through stamps becoming gummy and sticking together. Chickens kept in air conditioned houses increase in weight, lay more eggs. Candy companies use air conditioning in order to properly make certain candies. The uses are endless. We are amazed by the different utilizations of our units."