all contained some free phosphoric acid. Most phytic acid samples darkened with age and showed a progressive increase in the amount of free phosphoric acid. To be effective, it was necessary to add phytic acid prior to the deodorization process.

Evidence is presented to show that a large part of the synergistic effects of many activators can be explained by metallic inactivation. It is suggested that much of the antioxidant effect observed for rice bran, oat "flour," and cereal brans could be the result of metal inactivation by the phytic acid present in these materials.

Acknowledgment

The authors wish to express their appreciation to the several commercial firms who have furnished personnel, time, and equipment in performing tests and experiments on phytic acid; also to Robert Beal of our Engineering and Development Division for supervising the pilot-plant experiments and to the 18 tasters of the Northern Regional Research Laboratory's taste panel for their continued help and cooperation in the evaluation of edible oils.

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Steam Stripping of Lard[1]

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A STUDY was made to establish a means of evaluating the effect of steam stripping at high vacuum on removal of free fatty acids from lard. Normal industrial procedure is to supply steam through sparge pipes at the bottom of large vessels. The upward flow of steam bubbles provides agitation and stripping of volatile products. No data have heretofore been available to show the advantageous effect of adding mechanical agitation to this batch stripping procedure. Accordingly experiments were run, using rotating mixing turbines to disperse steam and to mix and agitate the batch thoroughly, also to determine optimum operating conditions of mixing for different rates of steam flow, pressure, and temperature. Stripping rates were increased as much as 50% with the use of mixing turbines. There was no difference in behavior between steam and nitrogen, hence steam acts as an inert gas in the stripping operation at the temperature involved.

Introduction

Oils and fats from natural sources contain small amounts of impurities in addition to the fatty glycerides. Included among these impurities are high molecular weight alcohols (sterols), hydrocarbons, free fatty acids, protein residues and other nitrogenous matter, phosphatides, and carotenoid pigments. These materials not only decompose into odoriferous substances but may act as catalysts, bacterial substrates, or inhibitors, either retarding or promoting reactions contributing to the spoilage of oils and fats (5).

Among the most abundant and difficult to remove of these substances is the free fatty acid, the presence of which in the oil is reported to cause an unpleasant taste, the so-called rancid taste. In order to eliminate this undesirable effect the concentration of free fatty acid in the lard must be reduced to less than 0.1% (1).

Countless methods have been employed in industrial processes to deacidify fats and oils including various means of neutralization by alkali and esterification, the removal of acids by extraction, steam distillation, and steam stripping. The steam stripping operation is the method most widely used in industry today for the deodorization of lard and hydrogenated vegetable oils. This stripping may be briefly described as the passing of steam through oil at a high temperature in a container in which a vacuum is maintained. This will effect the removal of not only free fatty acids but also all of the volatile impurities mentioned above. Although continuous steam stripping has been employed to a limited extent, the batchwise operation is by far the most widely used.

This investigation was undertaken to establish a means of evaluating the steam stripping effect under various conditions and to correlate the change in this effect with the variation in a given condition. The conditions varied were the amount of agitation by a mixing impeller, methods of introduction of the stripping steam, the absolute pressure in the stripping tank, the temperature of the liquid, and the rate of steam introduction.

A refined lard usually containing less than 1% free fatty acid was used, and a fresh batch was provided

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for all runs. High pressure steam was used for stripping with the exception of a few runs in which nitrogen was employed as the inert stripping gas.

Apparatus

The experimental apparatus consisted of a steam source and devices for measuring the rate of steam input, a steam ejector, and a vacuum gauge for maintaining and measuring the vacuum in the stripping system, a stripping tank with a sampling spigot and a feed line for introducing and eliminating the lard, an air motor for driving the mixing turbine in the tank, and a means of introducing hot nitrogen instead of steam for stripping.

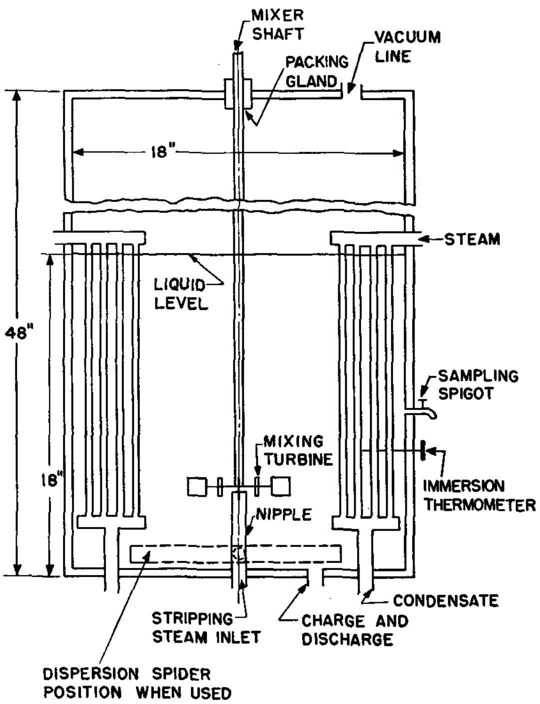


FIG. 1. Cross-section diagram of stripping tank.

The stripping tank, Figure 1, was an 18-in. diameter, 48-in. high, cylindrical iron pressure vessel with ½-in, thick walls and 1-in. thick flange heads at the top and bottom. It was equipped with four 4-tube baffles (½-in. tubes spaced one inch apart), placed in radial directions with respect to the center of the tank. These baffles served as heating and cooling tubes. Steam entered the top header of a baffle, and condensate was withdrawn through the bottom header. Two-inch permanent insulation covering the curved surface of the tank and 3-in. temporary insulation covering the flanges together with six electric strip heaters scattered over the surface of the vessel aided in maintaining constant temperature by minimizing heat leak.

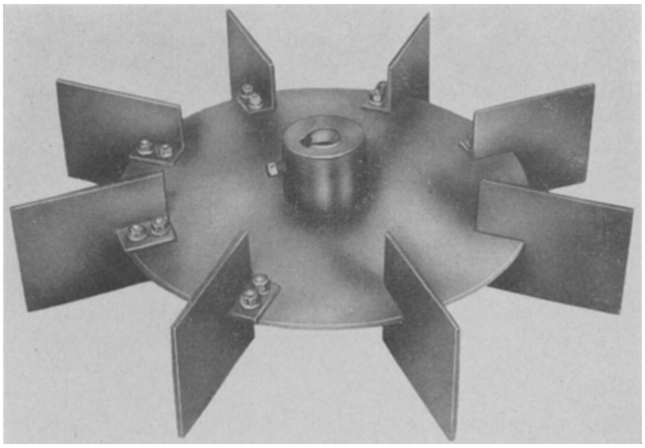
The lard was introduced and removed through a ⅜-in. tapped hole in the bottom flange, to which was connected the feed line. The stripping steam entered the tank through a ¾-in. tapped hole in the center of the bottom flange. The steam was distributed by one of two methods: the open pipe discharging just below the mixing impeller, or by a spider located below the impeller. Both are shown in Figure 1. The spider was made up of a side arm cross with four dispersing arms and a connecting nipple. The four arms were 6 in. long, ¾-in. nipples, capped at the far end and perforated with $\frac{1}{16}$-in. holes, one inch apart along two lines parallel to the axis of the nipple. These lines were 45° apart, and the nipple was set in a position such that the holes in one line faced downward and those of the other faced a line 45° to the vertical.

The high pressure input steam was available at 300 lb./sq. in. abs. to heat the batch and for sparging. The rate of steam introduced into the tank as stripping steam was determined by measuring the pressure drop across a $\frac{1}{16}$-in. diameter orifice. The orifice was calibrated periodically.

Vacuum was maintained in the system by a two-stage steam ejector, supplied with 90 lb./sq. in. boiler plant steam. The vacuum line was made up of 2- and 3-in.-diameter galvanized iron pipe, which required cleaning every two or three months. Traps in the line were emptied after every run.

Agitation and mixing was produced by means of a ½-hp. "Lightnin" air-driven motor mounted directly above the center of the tank and geared to a ½-in. diameter shaft. A six-blade, 6-in. diameter (tip to tip) flat-blade turbine (Figure 2) was attached to the end of this shaft so that the turbine was immersed in the lard and would rest one-third of the total liquid depth from the bottom flange as well as ½ in. above the nipple through which the steam was introduced.

When nitrogen was used instead of steam, it was introduced through ¼-in. tubing and ¾-in. pipe, heated electrically and its rate of flow determined by a $\frac{1}{16}$-in. orifice and manometer.



—*Photo, Courtesy Mixing Equipment Company, Rochester, N. Y.*

FIG. 2. Flat blade turbine.

Experimental Procedure

Before beginning a run, the steam ejector was turned on and a vacuum maintained in the stripping tank. This was done to prevent oxidation of the lard during the heating period and to provide a means of drawing in the sample batch.

Approximately one-third of a 50-gallon drum of lard was melted after weighing and sucked into the tank through a flexible rubber hose. This filled the vessel to a depth of 18-in., equal to the diameter of the vessel. The drum was reweighed to determine the amount of material removed from it. The average weight of charge was 130 pounds.

The lard was agitated by means either of the turbine or the steam sparging alone through the spider, or by the rotating turbine with steam fed beneath it. Steam was sent into the baffles and additional heat supplied from the strip heaters attached to the outside surface of the tank.

Stripping steam at 300 lb./sq.in. (417° F.) was passed through a needle valve with which its rate was controlled, then through an orifice, and finally into the tank. The pressure on the lard was maintained constant at between 10 and 40 mm. of mercury absolute. Since steam at 300 lb./sq. in. (417° F.), when expanded through a valve adiabatically to a pressure of 10 to 40 millimeters of mercury, will have a temperature of 320° F., it is evident that the stripping steam would be heated by contact with the lard. The cooling effect of the stripping steam required that the batch be heated by steam in the heating baffles or by the strip heaters on the wall to maintain temperature and to offset heat leak.

At set time-intervals samples were withdrawn into 250-ml. Erlenmeyer flasks by producing a vacuum in the flask greater than that in the tank. Before breaking the vacuum in the flask, the sample was cooled by immersing the flask in cold water and shaking it. Inaccuracy due to holdup in the sample line was avoided by taking large "dummy" samples in a special flask to flush the line before each true sample was withdrawn.

The temperature and pressure were recorded immediately after each sample was taken. If the lapse of time between samples was large, they were recorded once or twice in this interval in addition to the readings taken at the sample time. The steam rate and temperature were manually regulated in order to maintain constant conditions.

When the run was over, it was necessary to cool the lard while maintaining it under high vacuum to prevent its discoloration by oxidation. To do this, cold water was circulated through the baffles and the agitation continued as during the run. When the temperature of the lard was 215° F., the line from the ejector was closed and steam introduced into the tank through the spider or nipple, whichever was being used. The pressure in the tank increased as the amount of steam in the tank increased until it became greater than atmospheric pressure. When this occurred, the valve on the feed line was opened, and the lard forced through the hose into a product drum.

Procedure for Testing Samples

Approximately 30 ml. of sample were placed in a 250-ml. Erlenmeyer flask of known weight. The flask and sample were weighed on an analytical balance, and the weight of sample thus determined. A beaker of 95% ethanol was heated to boiling, and 72 ml. of this alcohol carefully poured down the sides of the flask, washing any lard which may have solidified down with it to the bottom. The flask was heated on an electric heater, and the contents, while hot, titrated with dilute NaCH (about 0.05 N).

The percentage of free fatty acid (FFA) was expressed as oleic acid, which was calculated from the following equation:

$$\%\ \text{FFA} = \text{ml. alkali} \times \text{N} \times 28.2 \times 100 \div \text{wt. of sample}$$

where N is the normality of the NaOH solution.

Usually less than one hour elapsed between the drawing and testing of the sample. However it has been found through experimentation that if the sample is solidified, it may be kept for several days without appreciably affecting the amount of FFA in it.

Theory

Means of Evaluating Efficiency of Operation. The free fatty acids, as has been mentioned, exist as a large percentage of the impurity in lard and are also among the least volatile of the materials which can be removed from lard by inert gas stripping. These acids consist of unsaturated and saturated long chain carboxylic acids, the greater part of which contain at least 16 carbon atoms in their chain. Since they are among the least volatile of removable substances and since their concentration can be easily determined by titrating the fat, the amount of FFA removed may be used to indicate the degree of steam stripping which a batch of lard has undergone.

However, in using the above analysis to compare the rate of removal for different batches, a constant volatility of the mixture of FFA must be assumed at a given temperature for all the lard used. The validity of this assumption was established experimentally during the course of the work for laboratory processed leaf and prime steam lards.

The Stripping Equation

Assuming the law of mass transfer

$$\text{B}\,d\text{f}^* = \text{M}\,(\text{f} - \text{f}^*)\,\text{A}\,d\theta' \qquad (1)$$

where

B = Weight of a single bubble of steam.

A = Area of a steam bubble.

f* = Fugacity of FFA in the lard which would be in equilibrium with the FFA in the steam bubble at any point during its rise through the lard.

θ′ = The time elapsed since the entry of the steam bubble into the lard.

f = Actual fugacity of FFA in the lard.

M = A constant

Raoult's law for the low concentration region

$$\text{f}^* = \text{pC} \qquad (2)$$

where

p = The vapor pressure of the FFA.

C = Concentration of FFA in the lard,

and the material balance for FFA leaving the lard at any instant is

$$-\,d\,(\text{LC}) = \text{C}'d\text{S} \qquad (3)$$

where

L = The amount of lard present.

C′ = The FFA concentration in the exit steam.

S = Amount of steam passing through the lard.

the equation

$$dc/d\theta = -\text{KC} \qquad (4)$$

may be derived where

$$K = \left(1 - e^{\frac{-MA\theta'_{av}}{B}}\right)(p/L\pi)(dS/d\theta) \quad (5)$$

θ = The time elapsed since the beginning of the stripping operation.

θ'av = The average time a steam bubble remains in the lard.

π = Total pressure above lard.

S = Pounds of steam introduced.

p = Partial pressure of the FFA in the steam at equilibrium.

When the variables of equation (4) are separated and integrated:

$$\ln Co/C = K\theta \quad (6)$$

where

Co = The initial concentration of FFA in the lard.

Equation (5) predicts that if lnC is plotted *vs.* θ a straight line of slope —K results. The value of K is therefore an indication of the effectiveness of the operation and is a function of all of the variables affecting the rate of steam stripping. Figure 3 is a semi-log plot of C *vs.* θ for several typical runs.

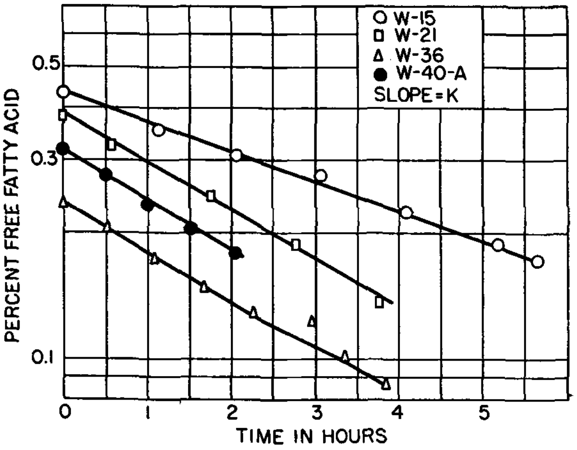


FIG. 3. Typical data. Change in free fatty acid with time.

Bailey (1) has derived a somewhat similar equation for a term efficiency E which is defined as

$$E = p'/p \quad (7)$$

where

p' = The partial pressure of the FFA in the steam of any instant.

The equation relating E to the operating conditions is

$$E = 1 - e^{KA\theta} \quad (8)$$

where

A = The interfacial area between steam and lard.

θ = The time of operation.

K = A constant.

Discussion of Results

It was proposed above that when all other conditions are constant, the rate of disappearance of free fatty acid is proportional to the amount of free fatty acid present. This leads to the theory that a straight line will result when the logarithm of the percentage of free fatty acid is plotted against the time of stripping, as expressed in equation (6).

In order to test the validity of this conclusion runs were made for various temperatures, using both the open tube with the mixer and the spider alone for distributing the steam. When the data were plotted as log percent FFA against time, straight lines were obtained. Several examples are shown in Figure 3. A summary of the data is tabulated in Table I.

In order to use the slope, K, of the line of the semi-log plot as a means of expressing the effect of operation, it was necessary to show that K would not vary for different batches of feed lard when all other operating conditions were the same. This, in effect, meant that the reproducibility of the data had to be demonstrated. Due to the fact that the vacuum produced by the steam ejector depended on the pressure of the steam supplied by the power house of the Institute (pressure varied from day to day), it was not always possible to reproduce the absolute pressure in the tank. Accordingly the effect of pressure was established first.

Effect of Pressure

The value of K is a function of the absolute pressure in the tank as can be seen from equation (5).

Assuming

$$d(MA\theta'_{av}/B)/d\pi = 0 \quad (9)$$

which is justified by the narrow range of pressures under consideration and by the fact that the effect of pressure on A/B tends to cancel out its effect on Mθ'av, the proportionality

$$K \propto \pi^{-1} \quad (10)$$

was postulated. This relation was used in correcting for uncontrollable variation in pressure from one run to the next, the range of variation of pressure being comparatively small. In Table I values of K are given for 18 mm. Hg. for comparison.

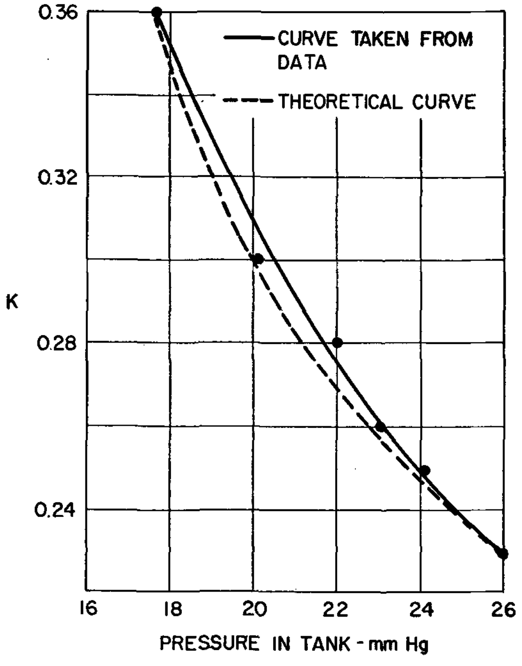


FIG. 4. Variation of rate constant K with pressure.

TABLE I

Some Typical Data and Conditions of Operation.
W indicates runs with spider alone. M indicates runs with mixer and open inlet.

Run No.	Temp.	Abs. Press.	Stripping Steam	Initial Acid Concentration	Final Acid Concentration	Total Time of Run	RPM.	K	K Corrected to 18 mm.
	°F.	*mm. Hg.*	*lbs./hr.*	*%FFA*	*% FFA*	*Hours*			
W-15	361	16	3.7	0.436	0.172	5.7	......	0.17	0.15
W-24	376	22	5.9	0.460	0.240	3.5	......	0.19	0.24
W-36	404	20	5.9	0.238	0.087	4.2	......	0.30	0.34
W-44A	398	18	4.9	0.287	0.144	2.0	......	0.31	0.31
W-46A	398	14	1.5	0.295	0.228	1.0	......	0.21	0.16
M-30	398	22	5.9	0.447	0.166	4.2	300	0.42	0.44
M-32	398	27	8.6	0.467	0.273	2.8	300	0.36	0.45
M-41	398	16	2.3	0.516	0.193	3.4	600	0.36	0.33
M-47	398	16	1.8	0.543	0.228	2.1	500	0.40	0.35
M-49	401	19	2.3	0.434	0.215	2.6	520	0.31	0.33

Figure 4 is a plot of K versus pressure for experimental data and for the theoretical curve. The curves are close and parallel and indicate that the errors introduced by using equation (10) should be very small for correcting for pressure variation in the range covered.

Effect of Temperature

From equation (5) it can be shown that K is dependent on temperature since p, A/B, and θ'av are all functions of temperature. Assuming that the volume of steam bubble is directly proportional to its temperature, then

$$A/B \propto T^{2/3} \qquad (11)$$

where T is the absolute temperature.

Also assuming that the velocity of rise of a bubble through the lard is proportional to the 1/2 power of the diameter of the bubble

$$U \propto 1/\theta'\text{av} \propto r^{1/2} \propto T^{1/6} \qquad (12)$$

where U is the upward velocity and r the radius of the bubble. The series of proportionalities was arrived at by combing (11) and (12) with (5) and using the approximation $e^x = 1 + x$ which holds for small values of x, giving

$$K \propto (MP/T^{1/2}\pi) \qquad (9) \quad (13)$$

Thus the plot of $K/T^{1/2}$ *vs.* T should result in a curve of the same shape as the true vapor pressure *vs.* temperature curve for the FFA in the lard if M does not depend on temperature.

Figure 5 is a plot of K [corrected to 18 mm. Hg. absolute pressure using equation (10)] *vs.* temperature, and Figure 6 is the value $KT^{1/2}$ *vs.* temperature. The latter curve does not approximate the shape of the curve of the vapor pressure of any of the common fatty acids, and therefore it is believed that values of $MA\theta'\text{av}/B$ are too large to justify the assumption of $e^x = 1 + x$ where $x = MA\theta'\text{av}/B$.

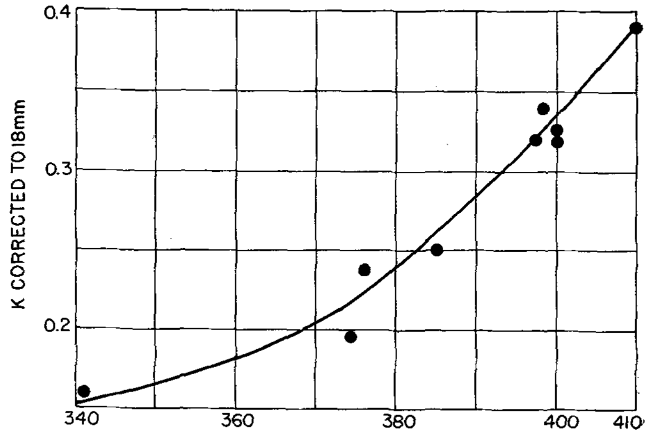


FIG. 5. Variation of rate constant with temperature.

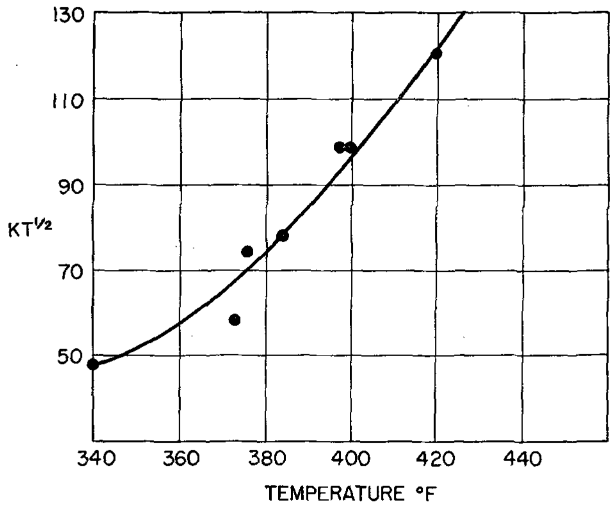


FIG. 6. Apparent vapor pressure vs. temperature.

Effect of Steam Rate

In equation (5) the relation between K and the steam rate, $dS/d\theta$, is complicated by the fact that M, A/B, and θ'av are all unknown functions of $dS/d\theta$ and would be very difficult, if not impossible, to evaluate separately.

Figure 7 is a plot of K *vs.* steam rate. It may be noted that a direct proportionality between K and steam rate was not observed. This serves to indicate that equilibrium was not reached between FFA in the exit steam and in the lard and that $MA\theta'\text{av}/B$ is not independent of the steam rate. The equation for the curve of Figure 7 is presented below in the section on Results, Equation (14).

Effect of Steam Distribution

In equation (5) it can be seen that as A/B and θ'av increase the value of K increases. Since θ'av increases as the bubble size decreases and since the ratio A to B is inversely proportional to the diameter of the average bubble, it follows that the smaller the size of the bubbles of steam rising through the lard, the greater is K.

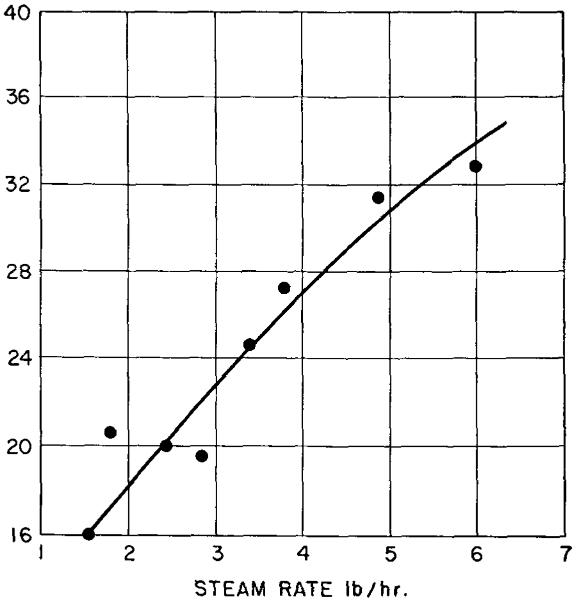


FIG. 7. Variation of rate constant with steam rate.

It has been common industrial practice to sparge steam into the oil by cross or other type spargers, depending on the jetting action to create small bubbles and to bring about some mixing due to the rise of the bubbles.

At high superficial steam velocities, such as those which are usually employed in the industrial process (about 6 ft. per second near the top of the liquid), choking occurs and the lard is lifted and splashed by the steam. This action causes a breaking up of many of the larger bubbles suspended in the liquid into smaller ones.

When dispersion of steam was accomplished with a turbine mixing impeller (Figure 2) instead of with the sparger as described above, it was found that a reduction could be made in the amount of steam required for the process. This reduction includes not only the difference in amounts going into stripping but also the reduced amount of steam required by the ejector to maintain the vacuum for a shorter time. This latter quantity is two to three times as great as the former. As an example, in an industrial deodorization operation, a 25,000-pound batch would use approximately 800 pounds of steam bubbled through the lard per hour, and another 2,100 pounds of steam would be used in the ejector each hour to maintain a vacuum of 12 millimeters of mercury. The mixer not only provides better steam distribution, but it also gives more agitation of the liquid.

Some research work has been reported by several investigators on gas distribution in a liquid using turbine agitation. Those giving performance data include the work of Cooper, Fernstrom, and Miller (3) on the oxidation of sulfite solutions by air for various power inputs, gas velocity, and sizes of equipment. Determination of air holdup in a cylindrical baffled tank filled with water, with air being introduced through a tube directly below the mixing impeller, was conducted by Foust, Mack, and Rushton (4). Sachs (6) investigated air holdup in oils of moderate viscosity, and Bartholomew *et al.* (2) give data for the fermentation processes for penicillin and streptomycin production.

In this work the performances of the spider mechanism and the mechanical agitator were compared for runs of various steam rates to determine proper conditions for large-scale design and to evaluate the economic feasibility of using mechanical mixing for steam stripping. Runs marked W in Table I are those where the sparger alone was used while those marked M are for use of the mixing impeller.

Results

Runs were made holding the lard temperature at approximately 398° F., pressure 18 mm. Hg., and the K's determined from the slopes of the lines resulting from lnC *vs.* θ plots of the data. A logarithmic plot of the slopes K *vs.* W, the steam rate, was made. Figure 8 shows the results and the straight-line correlations obtained.

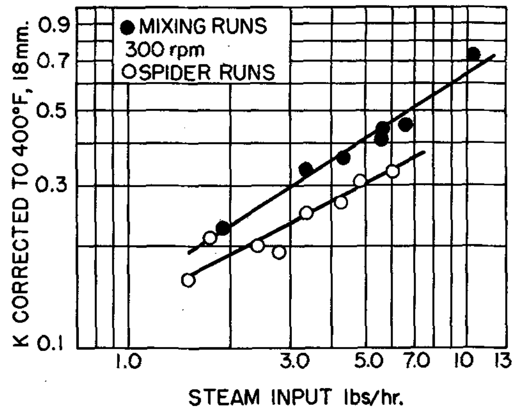


FIG. 8. Effect of steam rate on rate constant K.

Steam-flow rates in the tables and plots are shown as pounds of steam per hour for the total batch of 130 lb. Comparison of these results with large-scale equipment operation may be made by computing the rate of steam flow per pound of oil. For example when a steam flow rate of 800 lb./hr. is used with 25,000 lb. of oil in a commercial reactor, the rate is 0.032 lb./(hr.)(lb. oil). An equivalent rate in the experimental reactor would then be 130 (0.032), or, 4.15 lb./hr.

The equation for the line for distribution of steam by the sparger is

$$K = 1.3(dS/d\theta)^{0.53} \qquad (14)$$

Using the mixing impeller to distribute the steam and to mix the liquid, the solid point curve of Figure 8 was obtained for an impeller speed of 300 rpm. The equation for this line is

$$K = 1.5(dS/d\theta)^{0.65} \qquad (15)$$

It is obvious from a comparison of these curves that with an increase in mixing and distribution of the steam by the mixing impeller, it is possible to increase the rate of stripping by a value of 30 to 50% (vertical distance between the two lines) when a turbine speed of 300 rpm. is employed.

To test further the theory that the impeller will improve stripping rates by increasing the interfacial contact area between the steam and the lard, runs were made at 400° F., corrected to 18-mm. pressure and at a steam rate of 1.75 lb. per hour for various impeller speeds. (This corresponds to a low value for industrial use of 338 lb. of steam per hour for a 25,000-lb. batch.) The resulting data plotted in Figure 9, show the relation and benefit of using higher impeller speeds. Note that the value of K at zero rpm. is very close to that obtained when the spider mechanism is employed for distribution. Since the slope of the curve of K *vs.* rpm. should approach zero as equilibrium conditions are approached, it is clear that equilibrium conditions between steam and lard are not reached even at the highest impeller speeds used. For maximum steam economy the steam should be saturated with free fatty acids, but this was not attained.

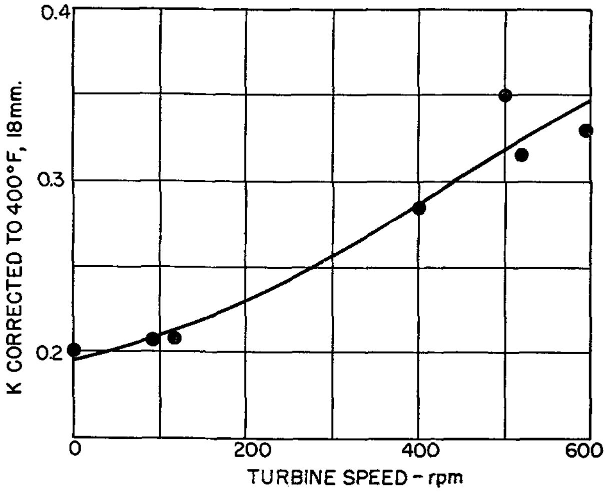


FIG. 9. Effect of mixing impeller speed on rate constant K.

Irregularity in Plots

In the semi-log plots of FFA concentration in the lard *vs.* time, deviations from the straight-line curves shown in Figure 3 were observed. The data sometimes resulted in irregular displacements of the curves as shown in Figure 10. In these cases, the rate K was taken as the slope of the first straight-line part of the curve.

In order to test whether these irregularities were due to a hydrolysis of fatty esters in the lard by the stripping steam, nitrogen was introduced as a stripping agent in place of steam. A plot of such runs shows similar irregularities indicating that hydrolysis was not the cause, and it is now believed that the effect is due to a condensation of FFA on the top or walls of the container, or in the vacuum line and periodic re-entrance of this FFA into the body of the lard.

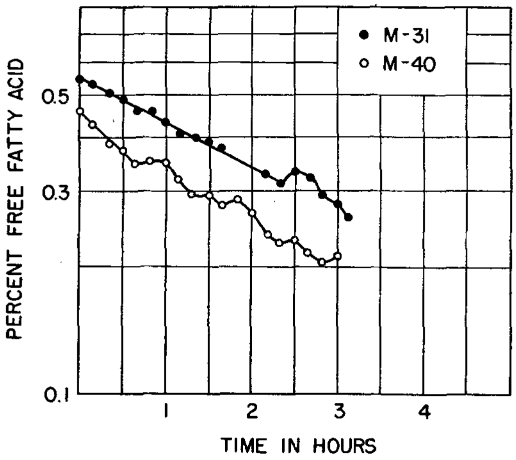


FIG. 10. Free fatty acid vs. time.

Conclusion

The rate of steam stripping of free fatty acids from lard can be evaluated in terms of a rate-constant K defined by Equations 5 and 6. Reproducible results were realized for many different batches of leaf and prime steam lards. The effects of temperature, total pressure, and steam rate were evaluated, thus making possible the calculation of stripping rate constants on a comparable basis. Comparable values of the rate constant K have been compared for conditions with and without the use of mechanical agitation with a flat-blade mixing impeller. Use of the rotating mixing impeller showed substantial increases (30 to 50%) in the reaction rate constant, over those obtained with steam sparging without the mixer. Accordingly the use of this type mixer should result in large savings of steam for stripping and for maintaining the necessary vacuum by the steam jet condenser.

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CORRECTION

A. L. Clingman of the South African Council for Scientific and Industrial Research, Pretoria, writes (as of March 13, 1953) that there was an error in the article by himself and D. A. Sutton, entitled "The Chemistry of Polymerized Oils. II. Dehydropolymers of Methyl Linoleate and Methyl Stearate," which appeared in the February 1953 issue of the Journal of the American Oil Chemists' Society. The footnote at the bottom of page 54 should have read as follows: "I.V. = 173.0; prepared by debromination of tetrabromostearic acid by the Hormel Research Foundation, Austin, Minn."