acted material (50.3 g.) was removed by distillation at 0.3-0.4 mm. pressure. The residue of condensation product (15.6 g.) had an iodine value of 63, a neutralization equivalent of 326, and contained 6.2% unreacted citraconic anhydride.

A mixture of 2.5 g. of this condensation product and 1.7 g. of a 69% aqueous solution of ethylene diamine was heated at 170°-180° for 20 hours. During this time the mixture was stirred by bubbling nitrogen through it. The product was a tough, tacky resin soluble in acetone and chloroform.

Condensation of Methyl Linoleate and Maleic Anhydride in the Presence of Dimethylaniline: Condensation was effected between 25 g. (1 mol.) of methyl linoleate and 8.3 g. (1 mol.) of maleic anhydride in the presence of 0.0%, 0.05%, 0.5%, and 5.0% of dimethylaniline. Percentages of dimethylaniline were based on the total weight of the reaction mixture.

In each case the reaction mixture was heated for 8 hours at 200° in an inert atmosphere. After cooling, the reaction mixture was filtered to remove a solid reaction product. The filtrate was distilled at reduced pressure to remove unreacted materials. Data on yields are given in Table II. Analytical data for the distillation residue are given in Table III. The solid reaction product was purified for analysis by washing with benzene. Table IV shows the analytical results. The solid product from the reaction containing 5.0% of dimethylaniline was insoluble in pyridine and the neutralization equivalent is not reported for this material. Saponification equivalents were obtained by heating the sample for 6 hours at 170°-180° with a 0.1 N solution of potassium hydroxide in diethylene glycol. Potentiometric titration was required because of the dark color developed.

Acknowledgment

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Summary

Adducts of methyl linoleate with itaconic and citraconic anhydrides have been prepared. Each adduct was separated into monomeric and polymeric components. With ethylene diamine, the itaconic and citraconic polymeric adducts gave gelled products at 170° in one hour and 3.5 hours, respectively. The adduct of citraconic anhydride and monomeric distillate gave a tough, tacky resin which did not gel during a reaction time of 20 hours at 170°-180°.

The effect of dimethylaniline on the condensation of methyl linoleate and maleic anhydride was studied. Increasing amounts of dimethylaniline were found to promote the formation of a heteropolymer and to decrease the yield of liquid adduct.

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Fatty Acid Distillation—Comparison of Two Column Types

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THE fractionation of fatty acids is an example of the increasing application of vacuum distillation. Although many papers have been published on the theoretical and practical aspects of petroleum fractionation, few have been directed to the theory, practice, and design of equipment for the fractionation of fatty acids. Equipment for fractional distillation of fatty acids has been described by Potts and McKee (12) and by Potts (11). Norris and Terry (8) treat the laboratory fractionation of fatty acids, and the literature on this subject has been reviewed by Bailey (1) and by Ralston (13). Vapor pressure data for saturated n-alkyl acids are given by Pool and Ralston (10), and Monick, Allen, and Marlies (6) give equilibrium data for lauric acid-myristic acid, methyl laurate-lauric acid, and methyl palmitate-methyl stearate.

In the study of distillation equipment design it is first necessary to have vapor-liquid equilibrium data for the materials being separated. It is the object of this paper to present the vapor-liquid equilibrium curves for lauric acid-myristic acid, myristic acid-palmitic acid, and palmitic acid-stearic acid, and to compare the performance of a bubble-cap column with that of a packed column with fibrous glass packing in separating these materials.

Equipment

Still. The still used for measuring the vapor-liquid equilibrium compositions was similar to that used by Gillespie (4). It is shown in Fig. 1. Heat is supplied to the material in the boiler by the heater, causing vaporization. The vapors entrain liquid, and the liquid and vapor remain in contact and come to equilibrium as they pass up the Cottrell tube to the disengagement chamber. Here the liquid and vapor are separated, the liquid returning to the boiler, the vapors passing to the condenser. The vapors are condensed and the condensed liquid is collected in the condensate trap, from which it overflows to the boiler. The stream of hot liquid separated from the vapor in the disengagement chamber is combined with the condensed vapor before its return to the boiler. Overflow from the condensate trap is returned to rejoin the liquid through a drop-counter and capillary; this gives a small hold-up and a constant rate of flow.

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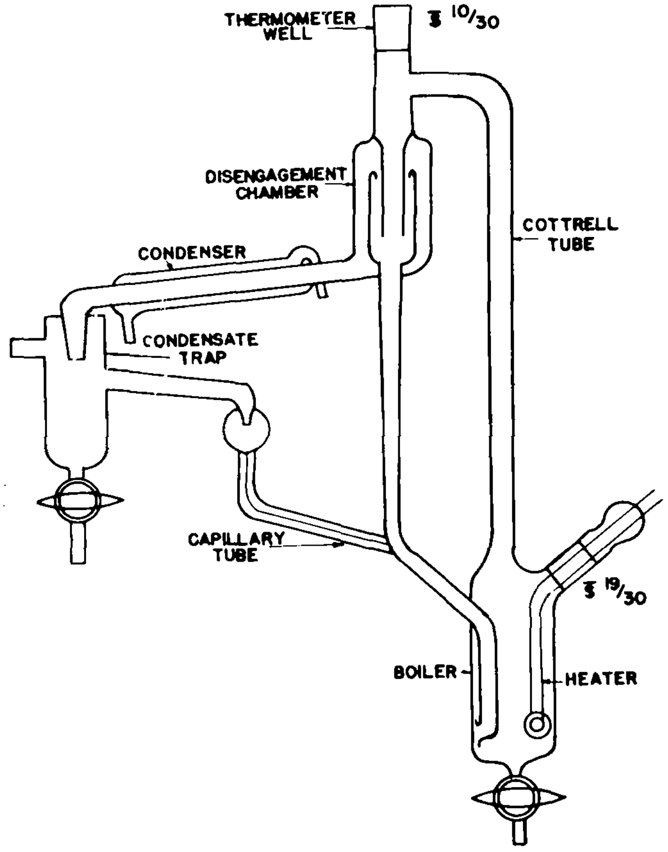

FIG. 1. Vapor-liquid equilibrium apparatus.

The apparatus was checked by measuring the equilibrium between ethanol and water at atmospheric pressure. The points obtained fell on the curve established by Carey and Lewis (2), which is one of the most accurate equilibrium curves available. To test for superheating at reduced pressures, the boiling points of pure water and pure ethanol were measured at several reduced pressures. The measured boiling points were in agreement with accepted values.

Packed Column. The packed column was made from 45-mm. glass tubing. It was connected by a ground glass joint to a one-liter flask which was heated by an electric mantle. Above the column was a reflux condenser which returned the reflux through a drop-counter. Total reflux was used. The condenser was connected to a vacuum pump which kept the pressures at the top of the column at four mm. of mercury absolute pressure.

The column was packed with fibrous glass for a height of two feet. The fibrous glass was weighed and then placed in the column according to the method described by Minar, Koffolt, and Withrow (5). The pads of glass were cut in one-foot lengths and inserted by sandwiching the pads between two sheets of stiff cardboard. The cardboard and glass were pushed down, and then the cardboard was withdrawn. This was done until the column was filled. The upper half was charged so that the planes of the bundles were at right angles to those in the bottom half. The column was packed until an apparent packed density of seven pounds per cubic foot was obtained.

The column was insulated with asbestos and wound with a nichrome wire heater. The heating was adjusted with a Variac control to prevent excessive condensation on the wall of the column. The pressure drop through the packing was measured with a manometer filled with n-butyl phthalate.

Bubble-Cap Column. A bubble-cap column 45 mm. in diameter was made from Pyrex glass. This column, shown in Fig. 2, consists of two plates spaced five inches apart, with a sample trap above the top plate and a liquid seal below the bottom plate. Each plate contains one bubble cap and a downspout, which is arranged to give an average liquid depth of three-quarters inch over the plate. Reflux was returned to the sample trap through a drop counter. A reflux condenser provided total reflux, and the pressure at the condenser was held constant at four mm. of mercury. The still was a one-liter flask heated by an electric mantle. The pressure drop across the two plates was measured by a manometer filled with n-butyl phthalate and attached to pressure taps as shown. The column was insulated and wound with nichrome heating wire.

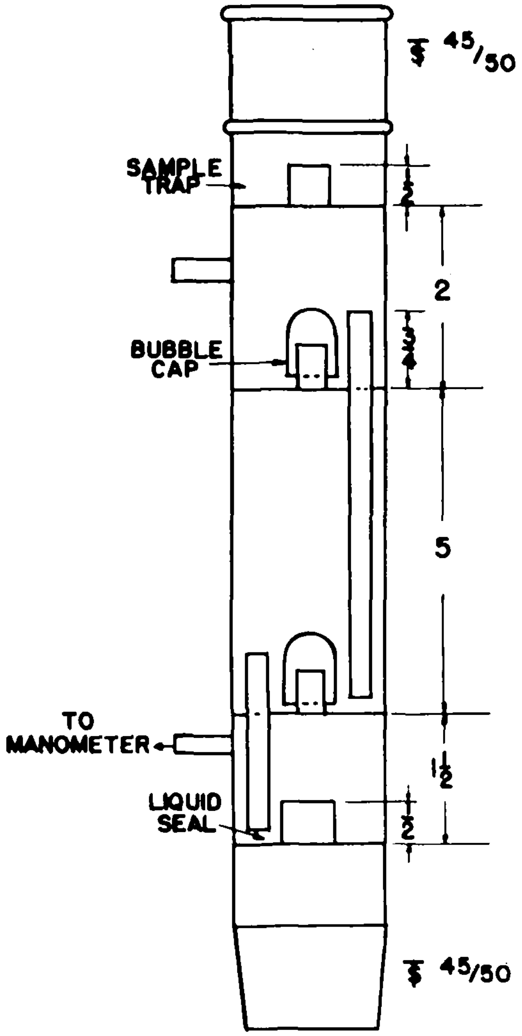

FIG. 2. Bubble-cap column.

Materials

Samples of four pure saturated fatty acids were prepared from commercial fatty acids of 90% purity. The commercial fatty acids were first fractionally distilled under reduced pressure, and a middle cut distilling at constant temperature was collected. The stearic acid was recrystallized from glacial acetic

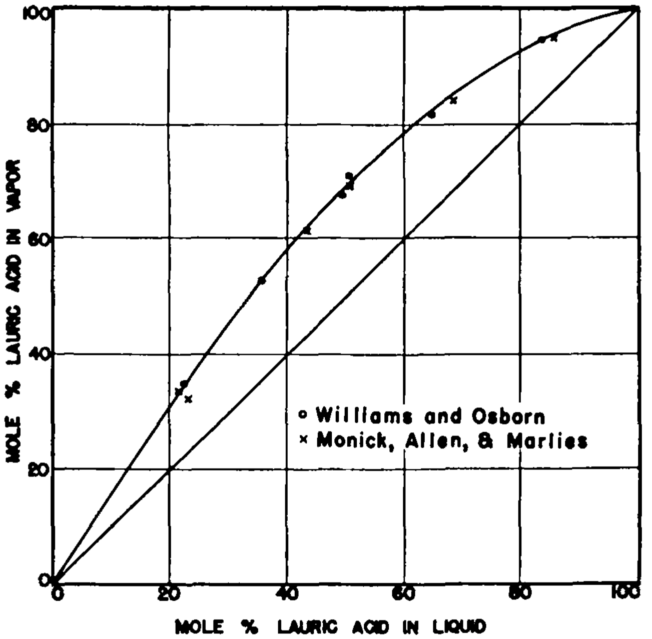

FIG. 3. Experimental vapor-liquid equilibrium data for lauric acid-myristic acid at four mm. Hg pressure.

acid several times and then from 95% alcohol. The other acids were recrystallized from benzene and 95% alcohol. Acids with the following properties were obtained:

Acid	Melting Point, °C.	Theoretical Melting Point	Acid Number
Stearic	69.2-69.7	69.6	197.3
Palmitic	62.5-63.0	63.1	218.4
Myristic	53.9-54.0	53.9	245.7
Lauric	43.5-44.0	44.2	280.1

These acids were used to make up synthetic mixtures for testing.

Analysis

Since the mixtures were known to contain only two different fatty acids, the analysis of a mixture could be made by means of the acid values. These were determined by titration of 1.5 grams of acid in 100 ml. of 95% alcohol with 0.1 N KOH. The indicator was two ml. of phenolphthalein. Because of polymerization in the still pot during the time required to reach steady conditions, it was necessary to make corrections on the acid values obtained. This was done in the manner described by Monick, Allen, and Marlies (6). In many cases samples of the liquid in the still pot were drawn off after three hours and again after four hours to determine the rate of change of the acid value. The average decrease in acid value was 0.4 per hour. A decrease of 0.7 per hour has previously been reported. The lower value may be due to the pre-treatment of the glass equipment before each run. The still was washed out with hot 50% sulfuric acid and then cleaned with alcohol or acetone and dried. This evidently removed the alkali from the surface of the glass, preventing the formation of calcium and sodium salts of the fatty acids, and thereby reducing ketone formation.

Equilibrium Data

The vapor and liquid compositions, and the boiling temperatures at four mm. Hg absolute are shown for lauric acid-myristic acid mixtures in Table I, for myristic acid-palmitic acid in Table II, and for palmitic acid-stearic acid in Table III. The x-y plots are shown in Figs. 3, 4, and 5. The data of Monick, Allen, and Marlies for lauric acid-myristic acid are shown in Fig. 3 and can be seen to be in excellent agreement with the data here reported.

TABLE I

Experimental Vapor-Liquid Equilibrium Data for Lauric Acid-Myristic Acid at Four mm. Hg Pressure

Temperature, °C.	Mole % Lauric Acid		Activity Coefficients	
	Liquid x	Vapor y	γ_1	γ_2
154	100.0	100.0	......	......
156	84.0	94.8	1.0	0.87
159	65.5	72.1	0.96	1.15
162	49.0	67.8	0.91	1.16
162.5	51.3	71.1	0.89	1.1
165.5	36.1	53.2	0.82	1.14
169	22.5	35.0	0.70	1.1
174	0	0	......	......

TABLE II

Experimental Liquid-Vapor Equilibrium Data for Myristic Acid-Palmitic Acid at Four mm. Hg Pressure

Temperature, °C.	Mole % Myristic Acid		Activity Coefficients	
	Liquid x	Vapor y	γ_1	γ_2
174	100.0	100.0	......	......
176.5	84.0	92.8	0.98	1.05
179	62.9	79.0	0.98	1.13
181	50.2	67.1	0.94	1.20
185	30.3	48.0	0.92	1.12
188	16.0	29.8	0.92	1.04
190	4.4	8.0	0.80	1.07
192	0	0	......	......

It is of interest to observe the deviation of these binary systems from Raoult's Law. Such deviation is commonly expressed as an activity coefficient, γ, which is defined by the equation

$$\gamma_1 = \frac{Py_1}{P_1x_1}$$

The vapor pressure data reported by Pool and Ralston (10) were used with the measured vapor-liquid

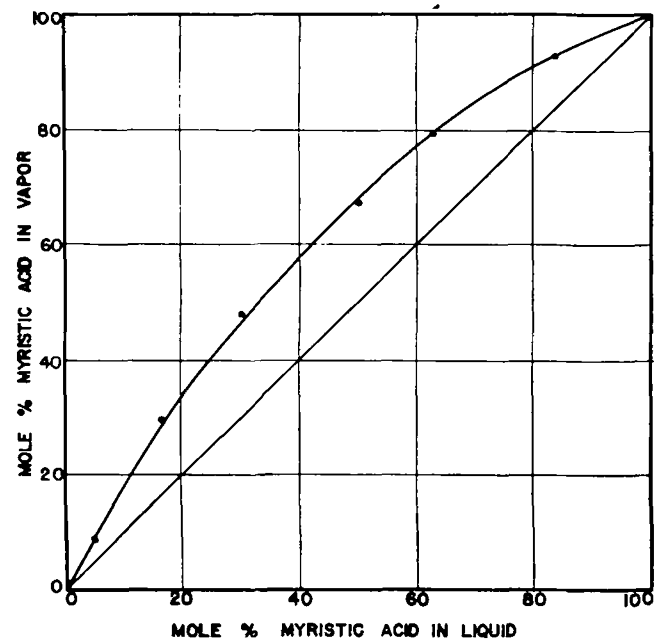

FIG. 4. Experimental vapor-liquid equilibrium data for myristic acid-palmitic acid at four mm. Hg pressure.

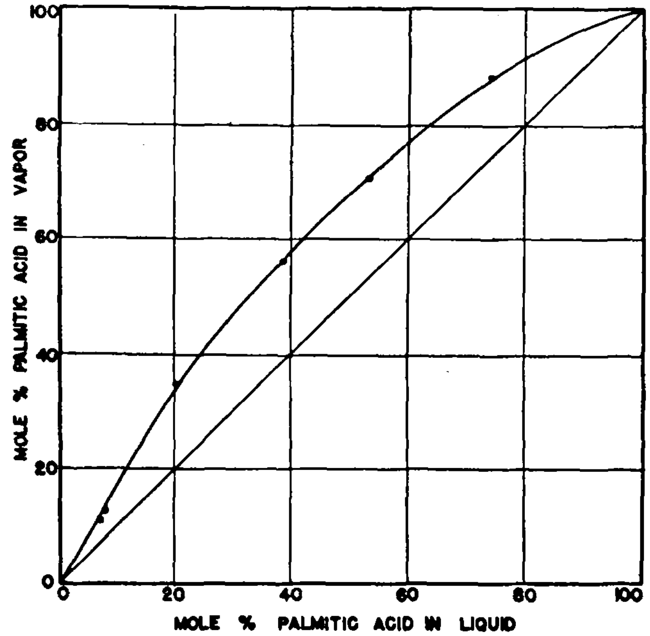

FIG. 5. Experimental vapor-liquid equilibrium data for palmitic acid-stearic acid at four mm. Hg pressure.

equilibrium data to calculate the activity coefficients, as shown in Tables I, II, and III.

Since the activity coefficient depends on the vapor pressure, which is in turn a function of the temperature, it is important to have reliable and accurate temperature data. The temperatures in these experiments were read to the nearest 0.5 degree C. Therefore, an activity coefficient in the range of 0.95 to 1.05 can be assumed to indicate an ideal solution.

All three systems studied show marked similarities. In all cases the more volatile constituent shows negative deviation from Raoult's Law in dilute solutions. There is no pronounced deviation of the less volatile component from ideal behavior. Another interesting characteristic is that the relative volatility of all three systems is approximately the same. The curves for myristic acid-palmitic acid and palmitic acid-stearic acid are superimposable while that for lauric acid-myristic acid is only a little different.

Fibrous Glass Packed Column

H.E.T.P. The packed column was used to distill various binary mixtures of the three fatty acid systems studied under total reflux. After steady conditions were reached, samples of the distillate and the liquid in the still were taken and analyzed. The number of theoretical plates required for the separation was calculated, and from this the height equivalent to a theoretical plate (H.E.T.P.). The still pot was assumed to be a theoretical plate. The effects of composition and vapor velocity on the H.E.T.P. were noted.

TABLE III

Experimental Liquid-Vapor Equilibrium Data for Palmitic Acid-Stearic Acid at Four mm. Hg Pressure

Temperature, °C.	Mole % Palmitic Acid		Activity Coefficients	
	Liquid x	Vapor y	γ_1	γ_2
192	100.0	100.0	......	......
194	74.0	88.2	1.1	1.07
198	53.0	70.1	1.02	1.1
201	38.1	56.8	0.98	1.1
205	20.0	34.9	0.93	1.06
207	7.5	12.5	0.79	1.05
207.5	6.5	10.5	0.76	1.04
209	0	0	......	......

TABLE IV

Distillation of Mixtures of Fatty Acids in a Fibrous Glass Packed Column

Mole Fraction in Still Pot	Mole Fraction of Distillate	H.E.T.P. in.	Vapor Velocity, ft./sec.	Pressure Drop, mm. Hg	Pressure Drop per Theor. Plate, mm. Hg
Lauric Acid-Myristic Acid					
0.24	0.49	30	4.5	12.6	15.7
0.26	0.52	30	4.9	13.3	16.6
0.48	0.78	26	5.2	13.4	14.5
0.55	0.81	40	7.2	15.1	25.2
0.70	0.91	40	3.7	11.9	19.8
Myristic Acid-Palmitic Acid					
0.23	0.54	30	7.3	14.4	18.0
0.25	0.60	26	4.5	12.7	13.8
0.30	0.57	48	7.1	13.9	27.8
0.35	0.62	30	4.7	12.6	15.7
0.50	0.79	26	5.9	13.1	14.2
0.55	0.82	26	5.1	12.7	13.8
Palmitic Acid-Stearic Acid					
0.18	0.48	28	2.4	11.2	13.1
0.22	0.50	34	6.5	16.9	23.9
0.37	0.68	40	5.7	12.3	20.5
0.56	0.79	60	8.1	18.7	46.7
0.59	0.84	40	4.0	11.4	19.0

The data for the three systems are shown in Table IV. The efficiency of the packing varies only slightly with vapor velocity and composition. However there is a large increase in the H.E.T.P. when the vapor velocity reaches approximately seven feet per second. From the pressure drop data it is believed that flooding began to take place at this velocity. For vapor velocities of from two to seven feet per second, the H.E.T.P. is fairly constant, and no correlation with vapor velocity is observed. The H.E.T.P. is also about the same for each of the three systems reported.

TABLE V

Variation of Pressure Drop with G/Φ Packed Column

Pressure Drop (In. water per ft. of packing)	G/Φ
Lauric Acid-Myristic Acid	
3.1	227
3.2	245
3.3	270
3.7	360
2.9	189
3.0	140
Myristic Acid-Palmitic Acid	
3.8	325
3.1	200
3.1	213
3.4	320
3.1	227
3.2	263
3.0	130
Palmitic Acid-Stearic Acid	
3.0	168
5.0	306
3.3	237
4.5	275
3.0	102

Pressure Drop. Pressure drop data for the fibrous glass packed column are reported in Table V. These data are shown in Fig. 6, in which log G/Φ is plotted against log ΔP. There is a break in the curve in the vicinity of G/Φ equal to 300. It is at this point that flooding is believed to take place. For the system palmitic acid-stearic acid this takes place at a vapor velocity of 6.3 feet per second; for myristic acid-palmitic acid at 7.1 feet per second, and for lauric acid-myristic acid at 7.3 feet per second.

Bubble Cap Column

Plate Efficiency. The runs with the bubble-cap column were made in the same manner as those with the packed column. The results were expressed in terms of overall plate efficiencies. The results are shown in Table VI. The efficiency of the column is low, varying from 25 to 40%. This is partly due to the plate design, but it will also be shown that a low efficiency is to be expected from the properties of the materials involved. The observed efficiencies are fairly constant for vapor velocities from 1.5 to 4.0 feet per second. Above four feet per second, the efficiency began to decrease. Entrainment became quite noticeable at about this velocity.

TABLE VI

Efficiencies for Separation of Fatty Acids in Bubble-Cap Tower

Mole Fraction in Still Pot	Mole Fraction in Distillate	Velocity, ft./sec.	Efficiency, %	Pressure Drop,, mm. Hg	Pressure Drop per Theor. Plate, mm. Hg
Lauric Acid-Myristic Acid					
0.20	0.42	3.0	40	2.7	3.4
0.30	0.59	3.7	35	3.2	4.6
0.40	0.69	4.4	32	3.5	5.5
0.43	0.75	2.2	40	2.5	3.1
0.55	0.84	2.4	35	2.7	3.9
Myristic Acid-Palmitic Acid					
0.49	0.74	3.9	35	3.2	4.6
0.52	0.78	3.1	35	3.2	4.6
0.26	0.52	5.1	25	3.7	7.4
0.22	0.50	1.6	35	2.3	3.3
0.58	0.80	4.3	30	3.2	5.3
Palmitic Acid-Stearic Acid					
0.26	0.58	2.4	40	2.4	3.0
0.22	0.50	3.2	35	2.6	3.7
0.45	0.74	2.4	35	2.5	3.6
0.56	0.79	6.0	25	3.5	7.0
0.18	0.42	4.8	28	3.2	5.7

Pressure Drop. The pressure drop per plate is recorded in Table VI. This varies with vapor velocity in a linear manner over the range studied. The height of liquid over the caps was estimated to be about five-eighths inch. The pressure drop based on this head of liquid would be 1.2 mm. of Hg per plate. Even at five feet per second however the pressure drop is small.

Discussion

Although it is recognized that results obtained on a glass column 45 mm. in diameter cannot be applied to the design of large-scale equipment, it is believed that some qualitative comparisons can be made. In addition, it is interesting to compare the results with certain design relationships which are used in equipment design. These concern the allowable vapor velocity and the theoretical plate efficiency.

The allowable vapor velocity in a plate column is given by the equation

$$u = K_V\sqrt{\frac{\rho_L - \rho_V}{\rho_V}}$$

This equation is discussed in Perry (9), who also gives values of K_V for various plate spacings and liquid seals. For a plate spacing of five inches and a liquid seal of five-eighths inch, which were the values in the experimental bubble-cap column, the value of K_V is given as 0.03. Using gas densities calculated by the ideal gas law, the allowable vapor velocity calculated from the equation is 4.4 feet per second. This agrees well with the conclusions reached from the efficiency data.

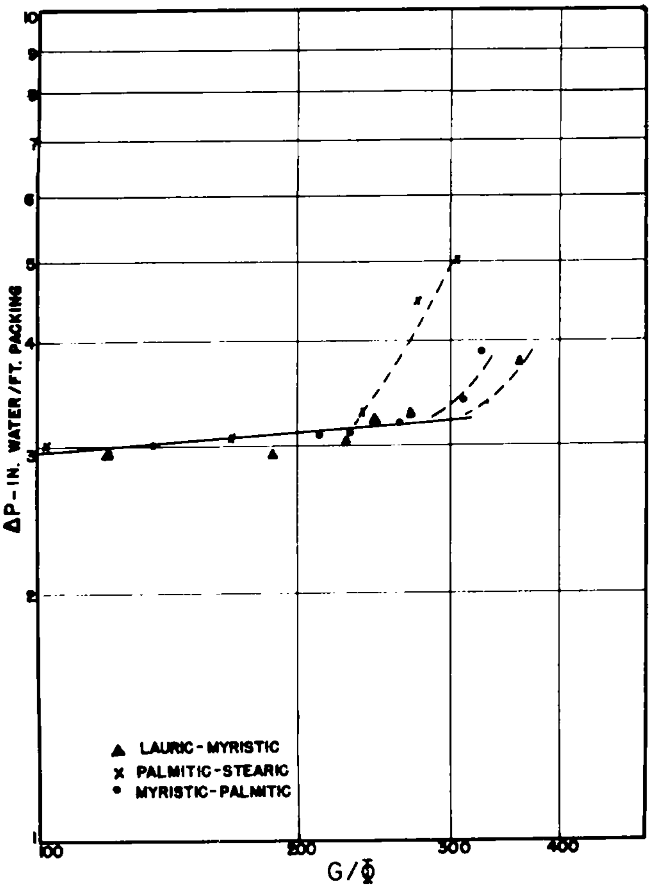

FIG. 6. Pressure drop versus G/Φ for fibrous glass packed column.

Attempts have been made by Geddes (3) and by Walter and Sherwood (14) to calculate the plate efficiency from the plate design and the properties of the liquid. The correlation of Geddes could not be used because of the lack of surface tension and density data at the boiling point. The correlation of Walter and Sherwood relates the Murphree plate efficiency to the viscosity of the liquid, the vapor solubility, the total pressure, the slot width, and the depth of the liquid on the plates. Because of the many assumptions made in the derivation of the equations, great accuracy is not claimed for the plate efficiencies calculated by this method, but it gives results which are in the right order of magnitude. Using extrapolated values for the density and viscosity, the plate efficiency for distilling a mixture of palmitic acid and stearic acid was calculated to be 20%. This includes several approximations, both of method and data, but it shows that the low efficiencies obtained experimentally for distilling fatty acids are what might be expected.

In a comparison of the packed column with the bubble-cap column the important factor is the pressure drop per theoretical plate. For distillation at low pressures, the pressure drop through the equipment may be several times the total pressure at the top of the column. If the pressure drop is high, the boiling temperature of the liquid in the still pot will be high, and an excessive amount of pitch formation will result. It is therefore important to keep the pressure drop as low as possible.

When the two columns are compared on the basis of pressure drop per theoretical plate, the data show that the bubble-cap column is more satisfactory in distilling fatty acids than is the fibrous-glass packing. A comparison of the data in Table IV with that in Table VI shows that for the same vapor velocity, the pressure drop per theoretical plate is about five times as great for the fibrous glass packing as for the bubble-cap column.

Although the allowable vapor velocity is lower for the bubble-cap column used, this should be improved by increasing the plate spacing. It was pointed out by Neureuther (7) that the H.E.T.P.'s on small laboratory columns are generally lower than on commercial columns. Therefore as the size of the column increases to commercial size, the H.E.T.P. and therefore the pressure drop per theoretical plate would be expected to increase. On the other hand, an increase in size of a bubble cap column might even increase the plate efficiency, due to better plate design and to liquid cross-flow. For larger sizes, the comparison should therefore be even more favorable to the bubble-cap column.

Conclusions

1. Vapor-liquid equilibrium data are presented for the systems lauric acid-myristic acid, myristic acid-palmitic acid, and palmitic acid-stearic acid at four mm. pressure. Deviations from Raoult's law are observed.
2. The performance of a small glass column packed with fibrous glass was studied. The H.E.T.P. was 30 to 35 inches and showed no consistent variation with composition. Above a vapor velocity of about seven feet per second, the H.E.T.P. increased.
3. The performance of a small bubble-cap column was studied. The overall plate efficiency was about 35%. The allowable vapor velocity was between four and five feet per second.
4. For the distillation of fatty acids at four mm. of Hg pressure the bubble-cap column was more satisfactory than the packed column containing fibrous glass since the pressure drop per theoretical plate was about one-fifth as great at comparable capacities. The comparison should be even more favorable to the bubble-cap column with larger sizes.

Nomenclature

G	pounds of vapor per square foot of cross sectional area per hour.
H.E.T.P.	height equivalent to a theoretical plate.
K_V	a constant.
P	total pressure, atmospheres.
p_1	vapor pressure of substance "1," atmospheres.
ΔP	pressure drop, inches of water per foot of packing.
u	allowable vapor velocity, feet per second.
x_1	mol fraction of substance "1" in the liquid phase.
y_1	mol fraction of substance "1" in the vapor phase.
γ	activity coefficient.
ρ_L	density of the liquid, pounds per cubic foot.
ρ_V	density of the vapor, pounds per cubic foot.
Φ	(density of vapor/0.075.)$^{1/2}$

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Evaluation of Peanut Protein for Industrial Utilization. A Review[1]

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Introduction

THE properties of peanut protein which are important for its industrial utilization as adhesives, sizes, water-thinned paints, and fibers are primarily physico-chemical in nature. To date the specific amino acid composition of the protein has not been shown to affect to any marked degree the industrial applications of the protein. The following discussion of the evaluation of peanut protein for industrial utilization is concerned therefore only with the physico-chemical characteristics of the components or fractions of peanut protein.

In 1913 Lichnikov (24) reported the separation and hydrolysis of albuminous substances from peanuts. The proteins were extracted from oil-free meal by means of water, 70% alcohol, 10% solution of sodium chloride, and 0.25% solution of potassium hydroxide in that order, thereby dividing the whole protein fractions into albumin, glutin, and globulin components.

In 1916 Johns and Jones (18-21, 23) reported and named the two major fractions of peanut protein, based on differences in solubility properties. They suspended 500 grams of oil-free peanut meal in 2.5 liters of a 10% solution of sodium chloride, clarifying the suspension by filtration. The protein fraction isolated by precipitation and filtration of the clarified solution by addition of ammonium sulfate to produce

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