Spaced Spiral Weave Metallic Cloth as a Column Packing Material for Stripping Soybean Oil

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PACKED columns, using such packings as Raschig rings and Berl saddles, are in common use in the steam stripping of solvent-extracted oils, such as soybean oil, for the removal of the last traces of the solvent. Efficient operation of the columns is desirable to obtain low steam consumption, together with effective removal of the solvent without heat damage to the oil. Investigations within the past 50 years have resulted in an increasing appreciation of the very pronounced effect of liquid distribution on the efficient performance of a packed column. Tour and Lerman (4, 5), in a study of liquid flow through a dumped packing, found that liquid introduced at the top of a column by a center-point feed source flowed down through a dumped packing in a manner conforming to the normal law of probability, or frequency distribution, providing the flow of liquid was independent of both redistribution equipment and the tower wall. The normal law may be expressed mathematically as:

$$Q_X = k\, e^{-h^2x^2}$$

where Q_x is the fraction of the total liquid starting down the center line of the packing that would fall into a trough of unit width located at a given distance down the packing and displaced horizontally a distance x from the center line. The factors h and k are constants which depend only on the packing height and on the type and size of the packing units; e is the base of the natural logarithmic scale.

In applying the normal law equation to experimental data, natural logarithms of the terms on each side of the equation may be taken to give:

$$\ln Q_x = \ln k - h^2x^2.$$

When the normal law applies, a plot of $\ln Q_x$ against x^2 will give a straight line having the intercept $\ln k$ and slope of $-h^2$.

Development of a Distributor Head

To determine the liquid distribution down through Raschig ring packing, an experimental tower, or column, 12 inches in diameter and 9 feet high was constructed from galvanized sheet iron. The lower end was flanged and bolted to a collecting grid which consisted of 4 concentric sheet metal rings 2 inches high, having diameters of 4, 6, 8, and 10 inches respectively, soldered to a 3/8-inch steel base plate 14 x 14 inches square in such a manner that their centers coincided with the center line of the tower. The tower wall served as a fifth ring, thus providing a total of five compartments. A 1/4-inch iron pipe nipple with 2 feet of attached rubber tubing was connected to each compartment as a drain. A 12-inch diameter piece of 4-mesh galvanized screen was placed on top of the partitioning rings to support the packing.

A constant-head tank was centered at the top of the tower to supply water at a constant feed rate. Flow rates from each compartment were measured with 250 or 500 cc. graduates and a stop watch. Data for tests employing center-point feeding and packed heights of 2, 4, 6, and 8 feet respectively are shown in Table I.

TABLE I

Distribution in Columns Packed With 3/4-Inch Raschig Rings

Packing depth, feet	Type of feed[c]	Flow rate, gal. per min.	Actual distribution as percent normal distribution[a]				
			Compartment no.[b]				
			1	2	3	4	5
2	C	0.765	458	193	85	16	4
	D	0.209	262	208	127	39	21
4	C	0.204	413	170	108	62	31
	D	0.201	202	174	148	63	30
6	C	0.197	207	168	120	73	38
	D	......	....	....	....	....	....
8	C	0.346	195	156	121	82	49
	D	0.223	128	137	122	95	64

[a]Average or normal distribution of total liquid over column area.
[b]Compartments numbered from the center outward.
[c]C—center feed. D—distributor feed.

Efficient operation of a packed column depends on a uniform distribution of the descending liquid over the surface of the packing. The data in Table I show

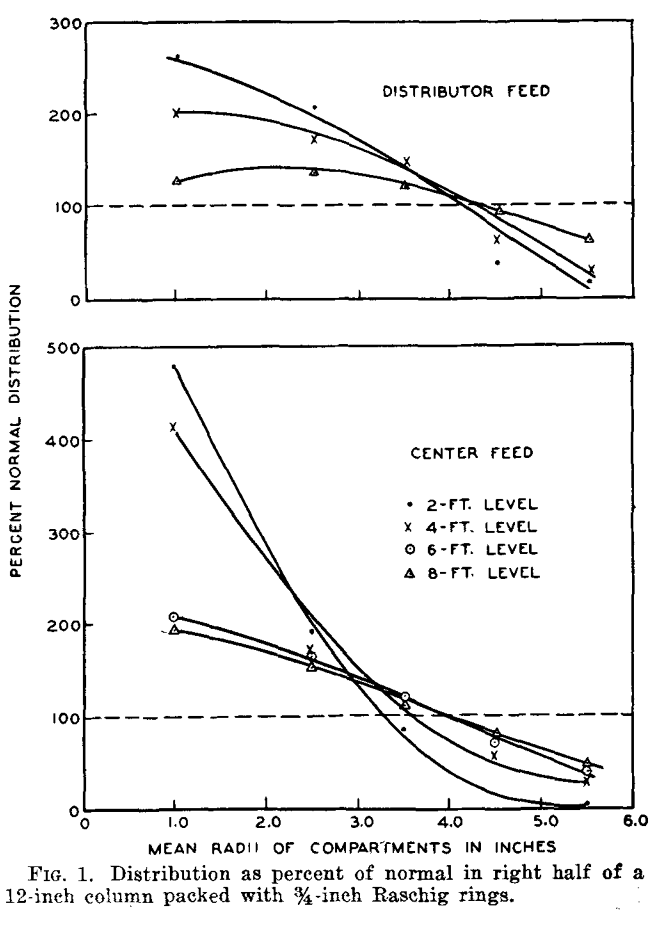

FIG. 1. Distribution as percent of normal in right half of a 12-inch column packed with 3/4-inch Raschig rings.

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that at a depth of two feet the concentration of liquid at the center (compartment No. 1) was 100 times that near the wall (compartment No. 5). The volume of packing covered in the top two feet of height was approximately that of a cone 12 inches in diameter by 24 inches high, or one-third that which would be covered in ideal distribution. Even at the eight-foot level the concentration at the center was four times that near the wall.

The variation from uniform distribution over one-half the column cross-section, that is from the center to the outside wall, is shown graphically in Figure 1, where uniform distribution is shown by the broken line. The average flow per unit area in each compartment has been recalculated as the percentage of the uniformly distributed flow, thus eliminating the variable of flow and allowing a direct comparison of the data for different packing depths. To determine whether or not the data conformed to the normal law of distribution as found by Tour and Lerman (4), logarithms of the percentages of total flows, Q_x, were plotted against the squares of the horizontal displacements, x, in Figure 2. The series of approximately straight lines obtained indicates reasonable adherence to the theory, as would be expected since this is a case of center feeding. These results indicated that it should be possible to increase the efficiency of a packed column beyond that secured by center feeding by the use of a suitable distributor.

The application of distributors used in various industrial processes to the feeding of a miscella (solvent-oil mixture) to the column was therefore studied. Circular spray heads and spray nozzles commonly employed in gas absorption equipment were found to plug if solids were present in the miscella. This plugging occurred also, but to a lesser extent, with a hollow cone spray nozzle. A splash plate was found to give very poor distribution over the column area.

Experiments with a slotted cone as a distributor showed that the slots were not receiving uniform quantities of liquid. Flat strips of metal were then attached to the surface of the cone forming radial partitions terminating at individual slots. The strips distributed the liquid uniformly to the slots, but no liquid flowed on the surface of the cone proper. These experiments led to the development of a distributor having no cone but consisting of 20 ⅛-inch rods attached to a bullet-shaped brass center and bent to form an umbrella shape. Partitioning rings of sheet metal were soldered to the upper edges of the rods to reduce any tendency of the liquid to fan across from one rod to another. This distributor head is shown in Figures 3 and 4.

The new distributor not only distributed a liquid with good uniformity over a column cross-section but also, because of the open type of construction, was not readily clogged by a liquid containing solids. When used with ¾-inch Raschig rings, results (Table I, Figure 1) superior to those with center feeding were obtained.

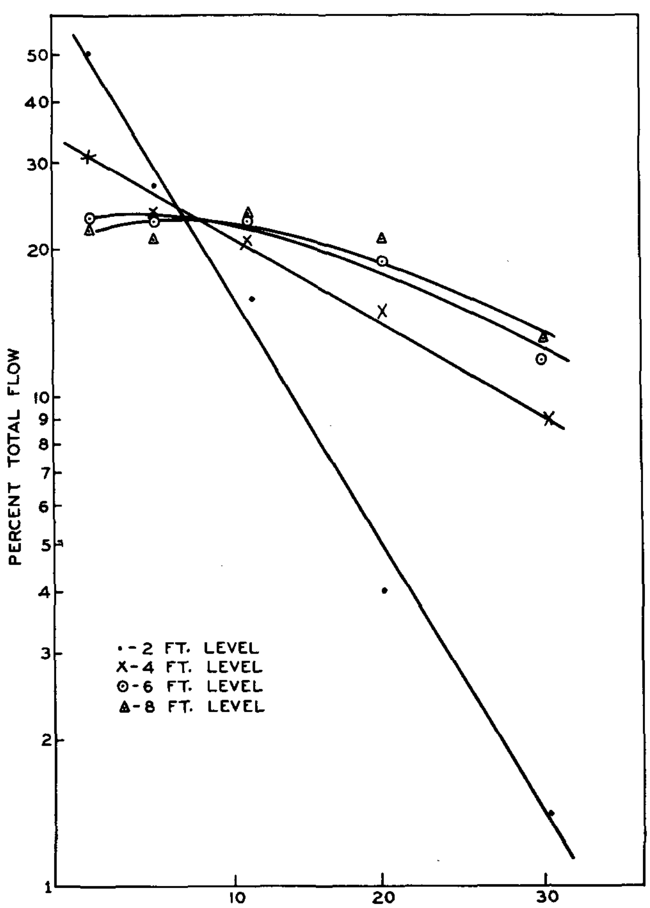

FIG. 2. Distribution in the right half of a 12-inch column packed with ¾-inch Raschig rings and with center feed. Relation of percent of total to square of displacement.

Evaluation of Stripping Column Performance

To evaluate the stripping performance of a packed column using the distributor head, test runs were made with a column used in the solvent recovery stage of a pilot plant designed for continuous solvent extraction of soybean oil. Since previous stripping studies (3) using this column had indicated somewhat better results with Berl saddles as packing than Raschig rings, the former were used. The arrangement of the equipment used is shown in the flow diagram, Figure 5.

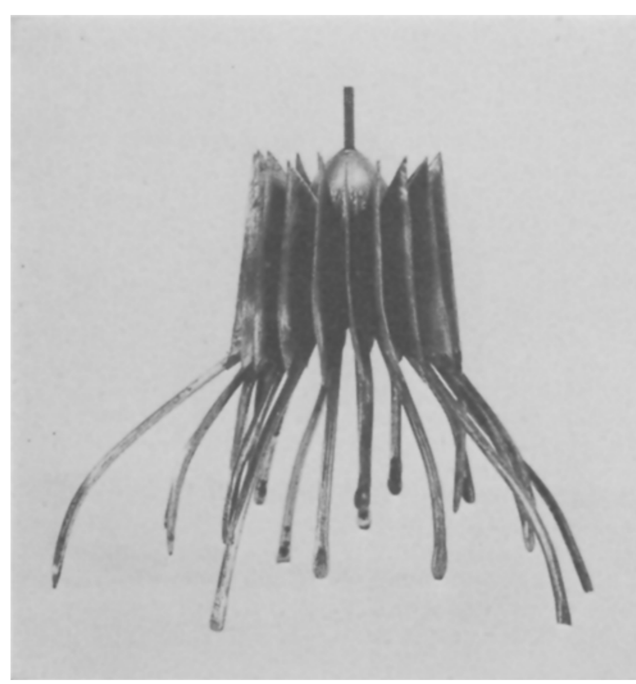

FIG. 3. Distributor head. Side view.

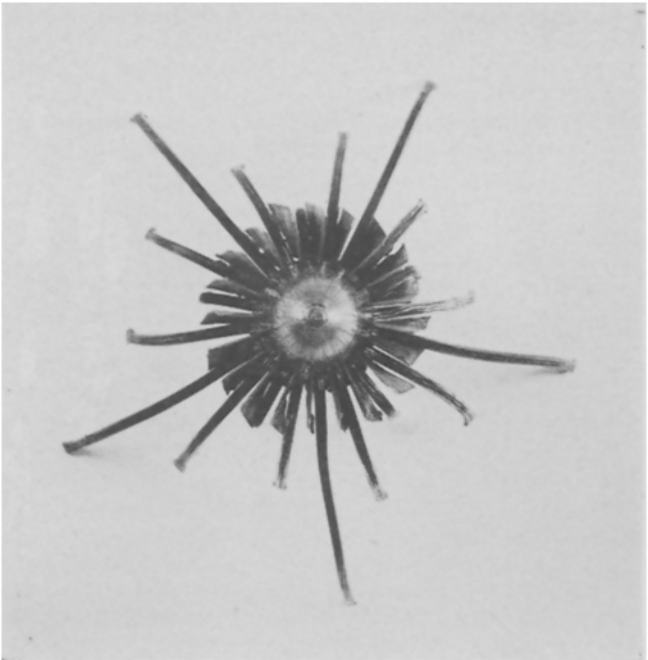

FIG. 4. Distributor head. Top view.

The column consisted of a standard black 8-inch iron pipe 9 feet in length, jacketed by a similar 10-inch pipe, and insulated with 2-inch, 85% magnesia insulation. Connections on the column were provided for the feed inlet, stripped oil outlet, and vapor outlet.

The feed, a miscella consisting of 80% soybean oil and 20% trichloroethylene by weight, was pumped

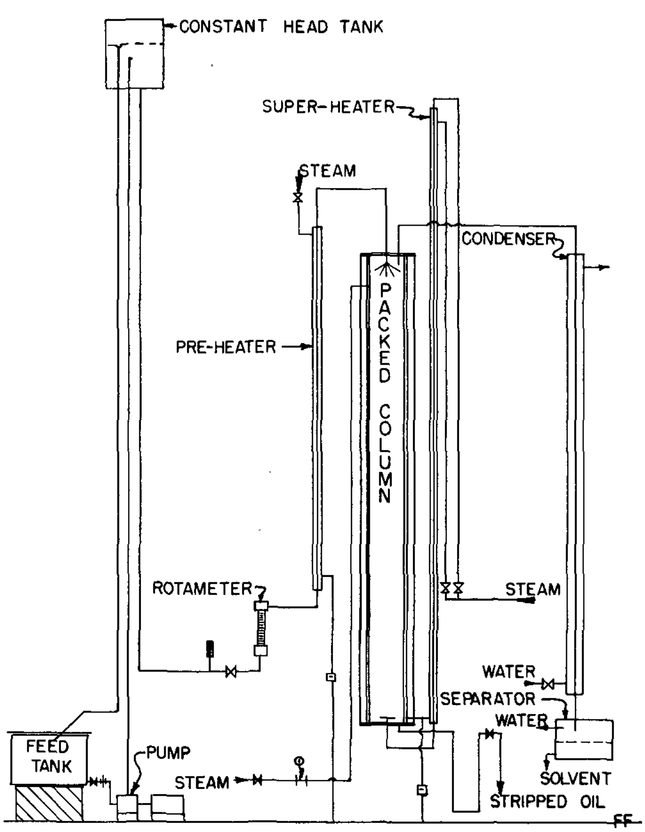

FIG. 5. Flow diagram of stripping equipment.

from the feed reservoir near the base of the column into a constant-head tank. From this tank the liquid fed by gravity through a rotameter to the top of the stripping column; initial distribution of the liquid was made by the distributor head shown in Figure 3. After flowing down the packing, the stripped oil was discharged through a gooseneck at the base of the column. Indirect steam was supplied to the column jacket at 80 lb. per sq. in. gauge. Additional live steam, improved in quality by means of a superheater, was introduced at the column base through a steam distributor having the form of a perforated cross. Vapors leaving the column were condensed and fed to a separator where the solvent was recovered.

Experimental operations were carried out in the following manner. The column was packed to a height of 8 feet with 1-inch Berl saddles by nearly filling it with water and slowly dropping in the packing units so as to obtain uniform packing. After draining the column, assembling the equipment, and allowing sufficient time for the apparatus to warm up, live steam was blown into the column and regulated to give the desired constant flow rate of condensate from the separator. Miscella was then fed to the column at a rate determined by the rotameter. The stripped oil was analyzed colorimetrically for trichloroethylene by a method described by Arnold and Hollowell (1).

Runs were first made with center-point feeding with various amounts of miscella and amounts of stripping steam to produce stripped oils containing the following percentages of solvent: less than 0.02, 0.02, 0.05, and 0.10. Feed temperatures, determined by thermocouples, varied from 100°C. at high feed rates to 120° C. at lower rates. Stripped oil temperatures varied from 140° to 150°C. Similar stripping runs were then made, using the distributor head. The data are presented graphically in Figure 6 showing the relation between the amount of oil stripped and the stripping steam required to reduce the solvent in the oil to a certain level and the limiting capacity of the column when producing stripped oil containing various solvent levels. Curves 1 and 3 show the increase in stripping efficiency and column capacity obtained by substituting distributor feeding for center-point feeding when stripping to a residual solvent content of 0.02%. The increase in stripping efficiency using the distributor head when stripping to 0.02% residual trichloroethylene was approximately 20%; in column capacity approximately 30%.

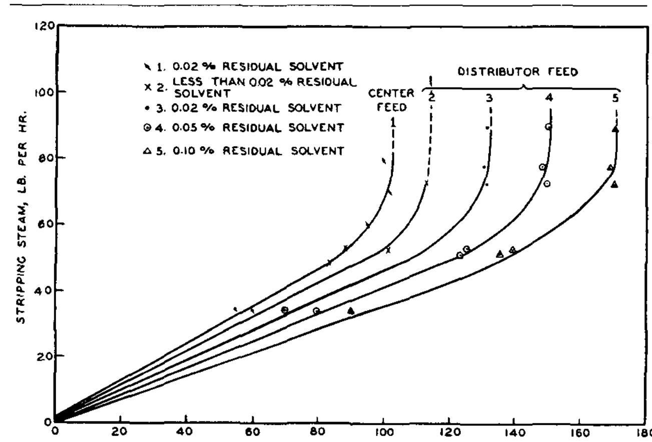

FIG. 6. Steam required for stripping soybean oil in column packed with Berl saddles.

Liquid Distribution Using Spiral Weave Metallic Cloth

The improvement secured in stripping efficiency by use of a distributor is limited since it merely sets the pattern at the top of the column; the distribution down the column is dependent upon the packing used. Certain types of metallic cloth appeared to possess filming properties which might adapt them for use as a column packing. Vilbrandt (6) reported that a column packed with a hose-knit cloth of flat copper ribbon showed slightly greater efficiency than a bubble cap column for distillation. Spiral weave metallic cloth of the type used for conveyor belts was investigated by Skow (2) as a tower packing for distillation and in this laboratory as a packing for a column used for stripping solvent from soybean oil. Excellent stripping capacities and efficiencies were indicated, but consistent results were not obtained. Distribution studies were therefore undertaken to determine the optimum conditions for the utilization of spiral weave cloth as a stripping column packing.

The material used was 10-mesh stainless steel spiral weave cloth with straight rod reinforcing (Figure 7) 26 inches wide rolled into a loose 8-inch diameter cylinder. The roll was inserted in an 8-inch sheet metal pipe 9 feet high, mounted on a collecting grid similar to that described for use with the 12-inch pipe except that it was equipped with 4 concentric circular compartments 2, 4, 6, and 8 inches respectively in diameter. Water was run onto the top surface of the roll by either center feed or by means of the distributor head.[2]

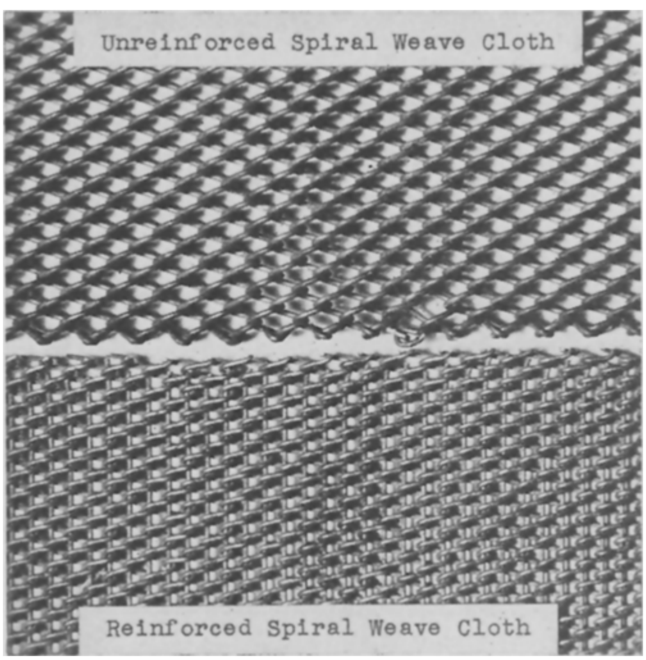

FIG. 7. Wire cloth used in the stripping studies.

The results, which are given in Table II, show that, when center feeding was used, most of the liquid remained near the center line of the column as it descended, none of it getting into the third or fourth compartments. Even with the distributor head feeding, the amount of liquid in the fourth compartment was only 55% of the normal amount for uniform distribution. The liquid actually moved toward the center of the column, just the opposite of the movement in the column packed with Raschig rings where the liquid tended to flow toward the column wall.

TABLE II

Distribution in a Column Packed With Unspaced Rolled Spiral Weave Wire Cloth 26 Inches High

Type of feed	Flow rate, gal. per min.	Actual distribution as percent normal distribution			
		Compartment no.			
		1	2	3	4
C	0.504	933	228	0	0
C	0.758	902	230	0	0
D	0.344	173	173	106	55

It appeared that the increase in liquid concentration at the center of the column was caused chiefly by the transfer of liquid from the outside to the inside of any given turn and thence across between turns to the outside of the adjacent turn on the center side. This transfer of liquid might result if the same amount of liquid were present initially on each of the two sides of the cloth so that the film on the inner or concave side would be slightly thicker than on the convex side, thus producing a tendency to flow towards the center of the column. Movement of liquid around the spirals which make up the cloth would tend to equalize the total amounts on the two sides of the cloth. It seemed probable that this transfer might be avoided by separating the layers from each other so they were not in contact. The use of some spacing device appeared to be the obvious solution.

Preliminary distribution studies using ¼-inch rubber tubing as spacing material showed that a continuous strip of material was unsatisfactory since the liquid tended to follow the tubing, spiralling towards the center. A spacing material large enough to prevent flooding across layers and of such nature as to prevent as much as possible the channelling of steam between layers was indicated. Three types of spacers were investigated: Raschig rings, Berl saddles, and balls. Rolled packing units composed of the spiral weave metallic cloth and the desired spacers were made by distributing the spacers on the surface of the cloth, laid flat on the floor, in such a manner that they were separated approximately ½-inch from one another. A sufficient length of cloth with spacers to form a cylinder which would fit snugly into the column was then rolled into a unit and secured with wire. A sufficient number of these units to give the desired height were placed one on top of the other in the column.

In addition to testing these in a column a simple visual test was devised in which a stream of water was impinged on a layer of the metallic cloth about three inches from the center of a vertically supported packing unit. The layers of cloth yielding streams of water at the base of the roll were checked to determine the horizontal displacement of liquid as it flowed down through the packing unit. This test was used as a check on all liquid distribution runs made in this study.

The results secured in testing the various spacing materials indicated that a spherical shaped particle used as a spacer produced the best liquid distribution

[2]When a change was made in column diameter, the rods in the distributor were readjusted to give proper distribution.

since it made only point contact between layers of metallic cloth. When using any spacer, there was always a better contact surface encountered between the inner surface of the cloth and the spacer than between the outer surface and the spacer. As a result, more liquid was displaced toward the inner layers than toward the outer portion of the rolled packing unit. When the roll diameter approached the diameter of the spacer, as it did near the center of the roll, this effect became more pronounced.

Liquid distribution tests were made with spacing materials ranging in size from ¼ to 1 inch, and it was found that ½-inch spacing materials were the most satisfactory in obtaining uniform distribution of liquid in a packing unit. Spacing materials less than ½ inch in size allowed liquid to flood across from one layer to the next at even moderate feed rates, and rolls spaced with materials larger than ½ inch gave less uniform distribution over a column cross section. Thus the ideal spacing material, from the standpoint of liquid distribution alone, appeared to be ½-inch balls. Representative distribution data on the three types of ½-inch spacers given in Table III and Figure 8 show the distribution effectiveness in the following decreasing order: balls, saddles, and rings.

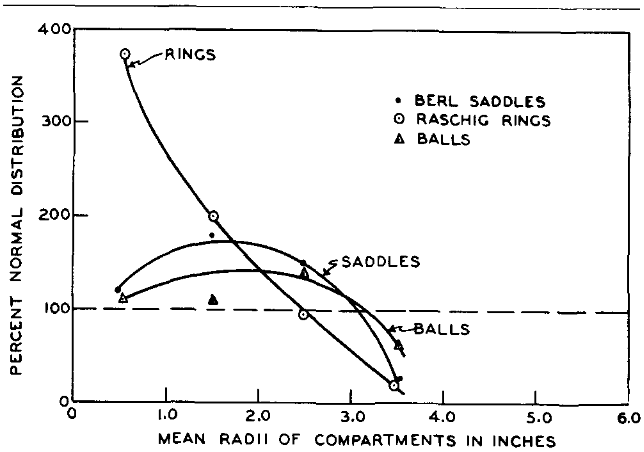

FIG. 8. Percent normal distribution in right half of column packed with spiral-weave metallic cloth spaced by Berl saddles, Raschig rings, and balls.

TABLE III

Distribution in a Column Packed With Spaced Spiral Weave Steel Cloth 52 Inches High With Distributor Feed

Spacing material	Flow rate, gal. per min.	Actual distribution as percent of normal distribution			
		Compartment no.			
		1	2	3	4
½" Berl saddles	0.272	104	180	160	18
	0.468	136	181	138	33
½" Raschig rings	0.464	372	202	96	20
½" Ceramic balls	0.235	112	110	146	63

In addition to the studies using the reinforced spiral weave cloth, distribution was determined on 8-mesh spiral weave cloth without the reinforcing rods (Figure 7) and with ½-inch Berl saddles as spacers. A plot of the data showed excellent distribution across the bottom cross section. Some distribution studies were made in which the distributor head was rotated at speeds between 30 and 50 r.p.m. The rotation decreased the tendency of the liquid to be picked up by the Berl saddles and displaced toward the center of the column. The distribution pattern changed less with changes in feed rate when the distributor head was rotated than when it was stationary.

Stripping Performance with Spaced Spiral Weave Wire Cloth

Stripping performance data (Figure 9) were determined for 10-mesh reinforced spiral weave cloth packing using ½-inch Berl saddles, ½-inch Raschig rings, and ½-inch ceramic balls as spacers. Stripped oil temperatures varied from 128° to 140°C. These data indicated that the Berl saddles rather than ceramic balls gave the best stripping efficiency and capacity. This somewhat better stripping efficiency and capacity of the saddles compared with the balls probably resulted from two factors: greater film surface and film movement on the saddles than on the balls and a better baffling of the steam by the saddles, resulting in a greater turbulence in the steam flow.

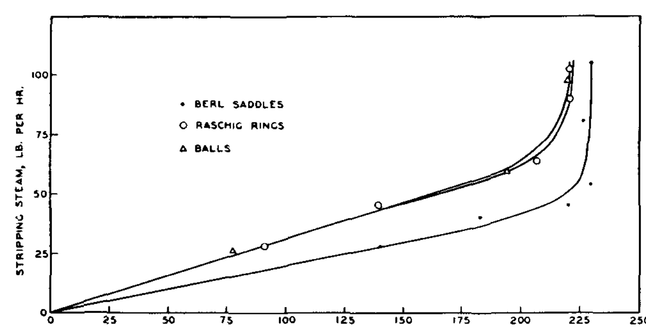

FIG. 9. Steam required for stripping soybean oil in column packed with reinforced spiral weave cloth spaced with Berl saddles, Raschig rings, and balls—residual solvent less than 0.02%.

Stripping data for non-reinforced 8-mesh spiral weave cloth with ½-inch Berl saddles as spacers are shown in Figure 10. Comparison of these data with data for the reinforced cloth (lower curve, Figure 10) indicated that the reinforced cloth was definitely superior since it gave greater vaporization efficiencies and an increased capacity. The relative efficiencies of the reinforced and non-reinforced spiral weave wire cloth are evidently not the result of differences in the distribution of the liquid across the column but rather the result of differences in the filming of the liquid down the cloth. Visual observation of the liquid passing down the two types of cloth showed that the liquid was flowing inside the spirals of the non-

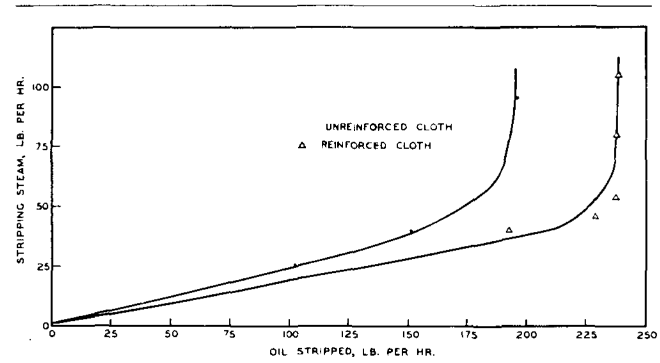

FIG. 10. Steam required for stripping soybean oil in a column packed with unreinforced and reinforced spiral weave cloth spaced with Berl saddles. Residual solvent 0.02%.

reinforced cloth and on the outside of the spirals of the reinforced cloth. Thus in the former case the stream of liquid was in a relatively thick film; and because of this and its position inside the spiral the film presented a smaller surface to the stripping steam.

A comparison of column capacities when stripping to less than 0.02% trichloroethylene shows an increase from 45 pounds per hour of stripped oil, when using center feed with 1-inch Berl saddle packing, to 101 pounds per hour using the same packing but distributor feed. The substitution of the reinforced spiral weave metallic cloth spaced with ½-inch Berl saddles increased the column capacity to 221 pounds per hour and reduced injected steam requirements to 0.21 pounds of steam per pound of oil. This improved behavior was obtained in a column with a spaced cloth packing of only 78 inches in height compared with a Berl saddle height of 96 inches.

Summary

A study of the effect of liquid distribution on the performance of a packed column resulted in the development of a distributor head for feeding which not only improved the distribution but decreased the steam consumption by about 20% and increased the column capacity by about 30% when stripping soybean oil-trichloroethylene miscella from 20% to 0.02% residual solvent. Liquid distribution studies with rolled packing units composed of 10-mesh reinforced spiral weave stainless steel cloth with ½-inch Berl saddles as spacers between turns showed that these units maintained a very uniform distribution of liquid down the stripping column. A column 8 inches in diameter with packing units having a total height of 78 inches had a capacity, when operated with the distributor head on 20% soybean oil-trichloroethylene miscella, for stripping 229 pounds of soybean oil per hour to a residual solvent content of 0.02%, using 0.20 pound of steam per pound of oil.

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The Effect of Trichloroethylene in the Hydrogenation of Soybean and Cottonseed Oils

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DESPITE the widespread use of hydrocarbon solvents for the extraction of oil-seeds there has been an increasing desire on the part of many processors to change to an essentially non-inflammable and non-explosive solvent such as may be found among the chlorinated hydrocarbons. This is mainly in the interest of safety. One large British mill changed over to trichloroethylene during World War II because of the danger of bombing, and results were entirely satisfactory. In this country the use of trichloroethylene in oil-seed extractions has been limited to a few plants processing soybeans. Further development has probably been hampered by the comparatively high cost of the solvent and, at least in the case of cottonseed, by certain technological difficulties requiring considerable research.

In addition to the foregoing, there has been a certain amount of misapprehension with regard to trichloroethylene as a solvent. An example of this is the belief held in some quarters that it is impossible to hydrogenate trichloroethylene-extracted oils because of residual solvent not removed during processing. This subject has been investigated in our laboratory and pilot plant, and we are presenting our findings herewith.

Laboratory-scale Hydrogenation

In some early experiments cottonseed oils containing 0.0-0.1% trichloroethylene (extreme levels were deliberately selected for testing so as to show up any effects) were subjected to hydrogenation in a small glass laboratory reactor,[1] in which hydrogen at atmospheric pressure is blown through an oil-catalyst mixture agitated by a high speed stirrer and maintained at any desired temperature. In this and all subsequent hydrogenations a commercial, reduced nickel catalyst containing approximately 25% metallic nickel was used. The catalyst concentration employed in the present experiments was 0.12%, and the reaction temperature was maintained at 165°C. (329°F.). Oil samples were prepared by adding various amounts of trichloroethylene to refined and bleached oil, mechanically expressed. In each test 190 grams were hydrogenated. Results are shown in Figure 1.

It is seen that oils containing .007% trichloroethylene hydrogenated as well as the trichloroethylene-free oil used for a control. Also it is apparent that oils containing as much as 0.1% trichloroethylene still hydrogenated reasonably well. The explanation for this is probably that trichloroethylene is continuously distilled from the oil under the conditions employed in this type reactor. It evidently does not form an irreversible adsorption complex with the catalyst. Reasoning from this, one would expect that the effect of any concentration of trichloroethylene in oil would vary with the operating conditions employed in the hydrogenation and, at least theoretically, oil containing any quantity of trichloroethylene could be hydrogenated under suitable conditions.

[1] Baker Instrument Company, Orange, N. J.