The Semi-Continuous Deodorization of Oils in All-Glass Equipment

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THE deodorization of oils has hitherto usually been accomplished, in the laboratory, in batch-type apparatus (1). When large samples are required or when the volatile products from a large quantity of oil are to be collected, it is advantageous to use a continuous procedure. Two all-glass deodorizers will be described herein which can be operated semi-continuously. The only limitation is the capacity of the oil receiver.

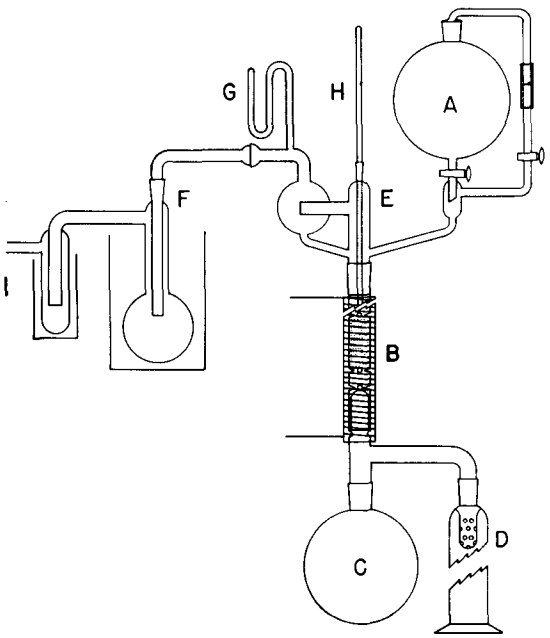

FIG. 1. Bubble-cap type, semi-continuous, glass, vacuum deodorizer.

The first of these, which is a bubble-cap type is shown in Figure 1. The oil reservoir A has a capacity of one liter. From it the oil flows through the drop counter, where an approximation of the rate of flow can be made. It then passes into Column B, which is 80 cm. long and 29 mm. in diameter and contains four bubble caps.

Figure 2 shows in detail one of the four units comprising Column B. Glass caps P, 45 mm. long and 16 mm. in diameter, are supported within the glass column by annular depressions Q and are prevented from rising more than about 4 mm. by indentations R in the column. Between each cap is a space 10 cm. in height filled with glass helices which are indicated by the dots S (Figure 2). These are held in place by indentations in the column.

In operation the oil flows down the column and seals the caps until they rise because of the pressure of the water vapor generated in vessel D (Figure 1).

The water vapor bubbles through the oil, and when the pressure has been reduced the cap falls back into position. As the cap oscillates, small quantities of oil pass by gravity into the next lower unit. In each unit the oil first accumulates in the upper helix-packed portion where it is agitated and scrubbed by the ascending water vapors. It then passes through the bubble-cap section as described. From the last unit the oil passes into the oil receiver C (Figure 1).

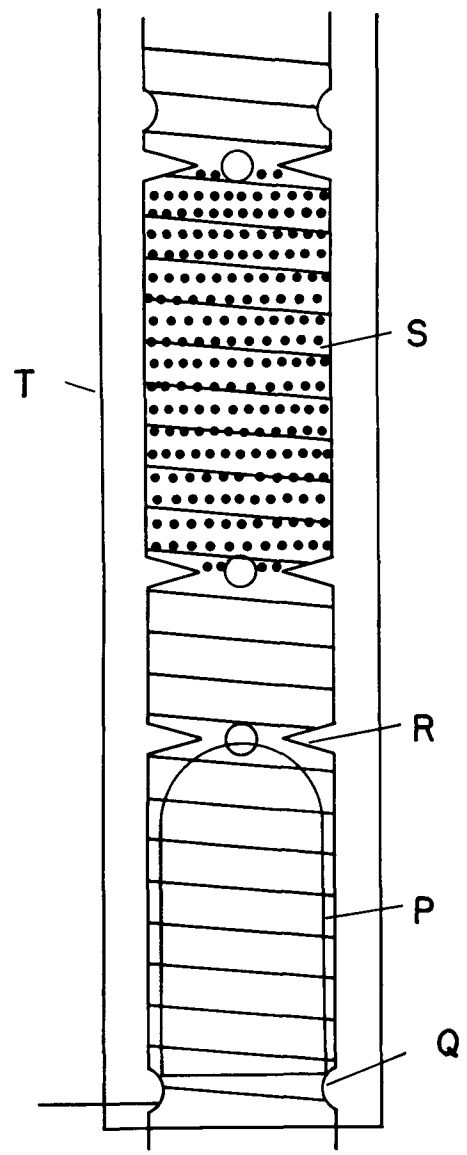

FIG. 2. Detail of one of the four units comprising column B.

The Column B (Figure 1) is encased in a glass tube wrapped with 30 feet of No. 26 resistance wire as shown. The temperature is controlled with a variable voltage regulator.

The temperature of the oil at the top of the column is registered by the thermometer H (Figure 1). The spray trap E prevents oil from being carried over

with the distillate, which is collected in dry ice traps F and I. A vacuum pump is connected to trap I, and the pressure is measured by the manometer G. The water vapor generator D is a 250-ml. graduated cylinder fitted with a baffle.

TABLE I

Oil Quality—Bubble-Cap Deodorizer

Oil Flow (g./min.)	Steam (% of oil)	Score and Standard Deviation	
		Oil	Control
6.8	1.1	8.6±0.45	8.2±0.71
7.5	1.6	7.8±1.26	8.5±0.45
8.0	1.7	8.3±0.32	8.3±0.32
11.0	1.7	8.0±0.30	8.1±0.32
15.0	0.5	6.7±1.34	8.5±0.45
23.0	1.2	4.7±1.05	8.3±0.32
No treatment		5.3±1.05	8.2±0.71

Table I shows the results of deodorization of a sample of reverted soybean oil in the bubble cap column at 0.45 mm. pressure. Taste tests were conducted by a trained taste panel in accordance with the procedure developed by the Northern Regional Laboratory (2). The oils were rated on a scale of 10, and the undeodorized oil was given a rating of 5.3 by the panel. The control used was a tested sample of bland commercial shortening. As shown in the table, oils deodorized in this column at a rate less than 15 g./min. were rated equal to the control. At a flow rate of 15 g./min. the quality of the oil was only moderately improved whereas at 23 g./min. there was very little improvement.

TABLE II

F.F.A. Removal—Bubble-Cap Deodorizer

F.F.A.	Oil Flow (g./min.)	Steam (% of oil)	% F.F.A.		% Removal
			Before	After	
Oleic	5.3	1.2	0.45	0.02	95.5
Stearic	6.0	1.3	0.52	0.04	92.5
Stearic	7.5	1.3	0.52	0.11	79.0
Stearic	24.0	0.25	0.52	0.46	11.5

Table II gives the results of deodorization, at 0.45 mm., of soybean oil containing known amounts of oleic and stearic acids. The free fatty acids in the samples were determined before and after deodorization. It will be seen that both oleic and stearic acids are almost completely removed at an oil flow rate of 6 g./min. or less. At 7.5 g./min. less stearic acid was removed, and at 24 g./min. very little was distilled.

It will be noted that the temperature at which the deodorizations were carried out has not been indicated. The thermometer H (Figure 1) indicates the temperature of the oil at the top of the column only, and this is affected by the influx of unheated oil. Accordingly, the deodorization temperatures were selected by experiment, and temperatures were duplicated by setting the voltage regulator at the same point on the scale.

The second of the two deodorizers is shown in Figure 3. It is known as a modified Oldershaw column-type deodorizer.

The oil reservoir A consists of a liter flask. The flow of oil into the evacuated system is controlled by the device N which consists of a stopcock, the bore of which is sealed at one end to form a cup. The stopcock is rotated at constant speed by the motor O. As the cup is rotated, it is alternately evacuated and

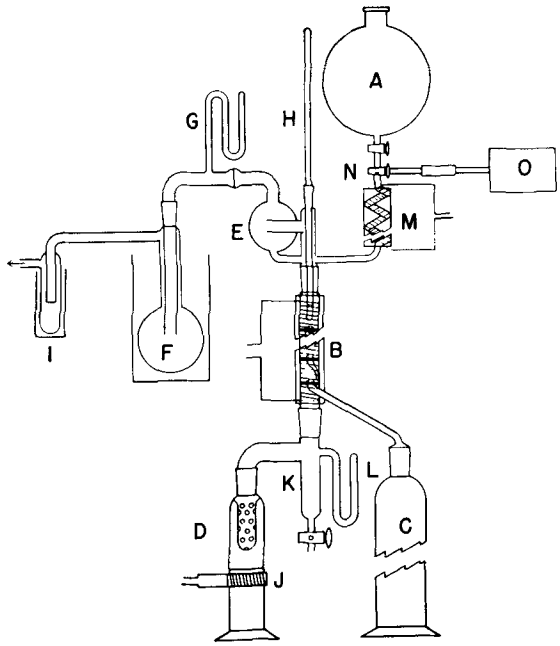

FIG. 3. Modified Oldershaw column type, semi-continuous, glass, vacuum deodorizer.

filled with oil from the reservoir. The oil is discharged into the preheater M without change in pressure.

The preheater consists of 40 inches of 7-mm. glass tubing in the form of a spiral. It is heated by a spiral wrapping of No. 26 resistance wire, and the temperature is controlled by means of a voltage regulator.

From the preheater the oil enters the modified Oldershaw column B, a part of which is shown in detail in Figure 4. The figure shows the two lowest plates, U and X, of a total of 15.

Each of the plates is perforated with many 0.5-mm.

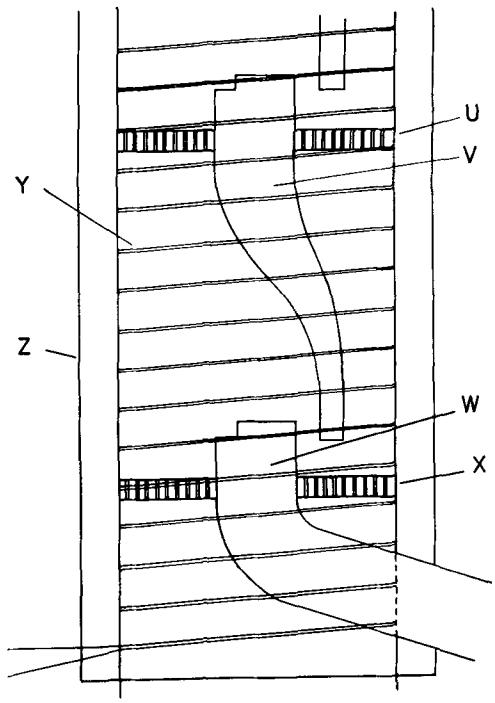

FIG. 4. Detail of two lowest plates of modified Oldershaw column.

holes and holds an overflow tube V (Figure 4). In operation the lower end of this tube is sealed by the oil accumulated on the plate X with the result that the water vapor must pass upward through the perforations and through the oil layer. The column is so modified that oil from the lowest plate drains into the oil receiver C (Figure 3). Each plate is 26 mm. in diameter, and the distance between plates is 30 mm.

The column is electrically heated by means of a heating element Y (Figure 4), composed of 30 feet of No. 26 resistance wire wrapped in a spiral on a close fitting glass tube enclosing the column. An outer jacket Z (Figure 4) of glass encloses the entire column and heating element.

The flow of water vapor is controlled by a small ring heater J (Figure 3), which is kept just below the water level in the water reservoir D. It is composed of a strip of sheet metal insulated with asbestos and wrapped with No. 26 resistance wire. The water reservoir consists of a 250-cc. graduate equipped with a baffle as shown. The trap K is necessary to catch the small amount of oil which drains from the column upon completion of deodorization. The vapor from the column passes through the spray trap E, into dry ice traps F and I. A vacuum pump is attached at I through a pressure regulator. Pressures are determined by means of the manometers G and L.

Temperatures are measured at the top of the column by means of the thermometer H and at any point on the column by means of a movable iron-constantan thermocouple inserted between the wall of the column and the heating jacket.

In operation the column is heated to the desired deodorization temperature, which is measured by means of the thermocouple. Then oil is allowed to flow through the preheater into the column, and the temperature of the preheater is varied until the thermometer and the thermocouple indicate the same temperature, showing that the oil is entering the column at the desired temperature.

A sample of the same reverted soybean oil used in testing the bubble-cap deodorizer was deodorized at 200°C. in the modified Oldershaw column deodorizer. The data are shown in Table III. A bland oil was produced at an oil flow rate as high as 17.5 g./min.

TABLE III

Oil Quality—Modified Oldershaw Column Deodorizer

Pressure (mm. Hg.)	Oil Flow (g./min.)	Steam (% of oil)	Score and Standard Deviation	
			Oil	Control
0.5	10.0	6.0	8.0±0.90	8.2±0.45
1.0	14.0	2.5	7.7±0.84	8.2±0.45
0.5	17.5	2.5	7.3±0.90	7.9±0.71

TABLE IV

F.F.A. Removal—Modified Oldershaw Column Deodorizer

Pressure (mm. Hg.)	Oil Flow (g./min.	Steam (% of oil)	% F.F.A.		% Removal
			Before	After	
0.7	23.0	2.0	0.52	0.08	85.0
4.5	7.2	8.7	1.51	0.32	79.0
9.0	5.5	10.0	0.45	0.13	71.0
7.0	7.5	6.6	1.51	0.54	63.5
3.3	24.0	2.0	0.45	0.26	42.0
3.5	21.0	2.0	0.45	0.32	29.0

Table IV shows the percentage of free stearic acid removed in the modified Oldershaw column deodorizer at 200°C. It is obvious that the best results were obtained at low oil flow rates or at low pressures. The rate at which free acid is removed is indicated by the data recorded for the second sample. Seventy-nine per cent of the free fatty acid was removed in one pass through the column even though there was an extraordinary high percentage (1.51) present in the original oil.

Summary

Two semi-continuous all-glass vacuum deodorizers have been constructed and tested. One operates on a bubble-cap principle. The other is constructed from a modified 15-plate Oldershaw distillation column. Both columns effectively deodorize oils and remove fatty acids, but the latter has the higher capacity.

Acknowledgment

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Phase Relations in the Solvent Winterization of Molecularly Rearranged Peanut Oil and Cottonseed Oil

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ONE of the major problems in the solvent winterization of cottonseed and peanut oils has been the production of more easily filterable crystals. The filtration behavior when hexane is the solvent is very unsatisfactory, especially for peanut oil. It has recently been shown that improved crystals can be obtained from acetone and from a solvent mixture consisting of 85 parts by weight of acetone and 15 parts of hexane and that these solvents have other advantages over hexane (2, 3, 7). Preliminary short-run pilot plant experiments by Holzenthal *et al.* (5) in this laboratory indicated that the crystals formed in the solvent winterization of cottonseed oil in this acetone-hexane mixture had excellent filtration characteristics.

According to Bailey and coworkers (1), much better crystals are obtained in the solvent winterization

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