A Laboratory Deodorizer

J. HEIDE-JENSEN, Aarhus Oliefabrik A/S, Denmark

Abstract

A laboratory deodorizer is described that has rapid heating, safety and convenience in operation, and a high degree of reproducibility. The range of capacity is from 50–2500 g.

Introduction

NOWHERE besides the edible oil industry has the unit operation of deodorization, i.e., steam stripping or distillation under vacuum, been developed to such perfection. Economy of steam and exclusion of air have been especially developed. Since deodorization was started in the Nineties (1) the most reliable equipment was found in the plant. The laboratory units were usually not leak-proof. However, introduction of the steam generator, published by Dutton and Schwab (2), permitted oil deodorized in the laboratory, to become models or standards of high quality for the plant. Dutton and Schwab combined four deodorizers into one oil bath and connected them to one condenser. We desired to have: 1) independent and sturdy single units, 2) more rapid heating, 3) omission of oil bath, and 4) increased safety and convenience.

Apparatus

The design of the apparatus is shown in Figure 1 and one of the units is shown in Figure 2. At the top is the steam generator with the steam tube extending to the bottom of the deodorizer flask. The exhaust tube has a spherical ground glass joint connecting it to the condenser and to the mechanical vacuum pump. The manometer is connected to the exhaust pipe.

The deodorizer flask is supported by asbestos attached to the electrical heating coils. The oil is protected from light by a shade around the exterior glass parts and by the aluminum housing (cover and cylinder). This protects the operator if breakage of the flask occurs during operation, but breakage has not happened in more than 5000 runs. Underneath the heating coils is a swivel damper, which is closed during the heating period and opened during the cooling cycle, allowing a vigorous air stream to pass upwards from the 90 watt ventilator.

The steel heating coil consists of 2 × 450 watt elements that heat 2500 g of oil to 180C in 30 min and the oil is cooled by the air stream to 50C in 45 min. The current to the heating elements is controlled by a thermo-regulator-thermometer placed in a thermometer well sealed into the deodorizer flask. When the charge of oil is less than 200 g it is preferred to place this thermometer in an oil bath in which the deodorizer flask is immersed.

Deodorizer flasks of 250, 500, 1000, 3000, and 5000 ml have capacities from 50 to 2500 g of oil. The preferred total heights are 300 mm for the sizes up to 1000 ml and 400 mm for the two largest sizes. All the flasks have a B29 ground glass joint (length 32–35 mm).

The steam generator contains 120 ml distilled water which, on evacuation of the system, boils at about 10C below room temperature. The velocity of steam is controlled by regulating the distance between the 150 watt radiator and the glass. The evaporator is leak-proof as the stopcock is safeguarded by water-seals, not only around the plug, but also in the side-arm. This serves for release of vacuum.

During cooling, water must not enter into the oil; hence the ball-shaped top of the evaporator and the horizontal inlet to the steam tube. The inside diameter of the tube is 8 mm, but the opening of the outlet is only 2.5 mm.

When citric acid has to be added after the deodorization, glass parts having a side arm at the ground glass joint on the steam tube can be used according to Dutton & Schwab, but for routine work we prefer to omit an inlet at that joint.

The exhaust-tube is slightly inclined towards the condenser. The 20 mm inside diameter insures a sturdy construction and a relatively low speed of the water vapors (about 15 m/sec, when 15 g H_2O/h).

The condenser is a 2-liter round-bottomed flask with a cold finger (50 mm inside), containing solid carbon dioxide and ethyl alcohol. The flask is insulated, and the container is placed on a small, round table which may be adjusted vertically to fit the two different heights of deodorizer flasks.

Operation

Before the oil reaches higher temperature it is important to insure complete removal of air from the water and oil. This removal is achieved after less than 10 min of deodorizing at low temperature of

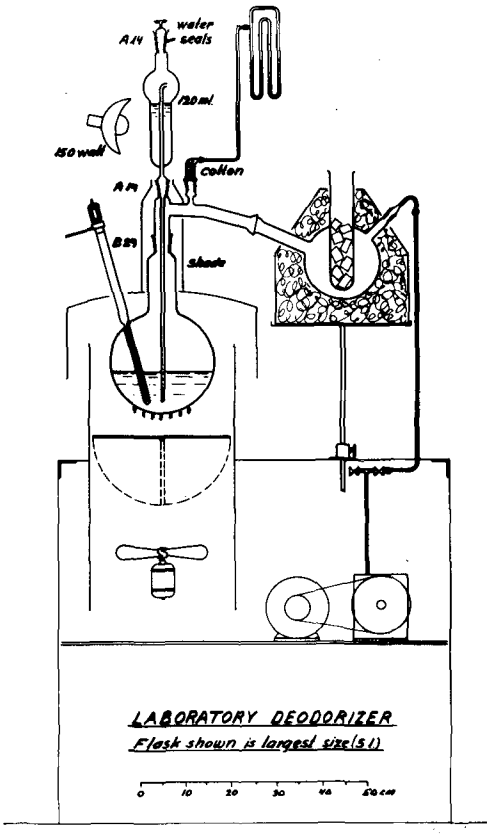

FIG. 1. Design for deodorizer.

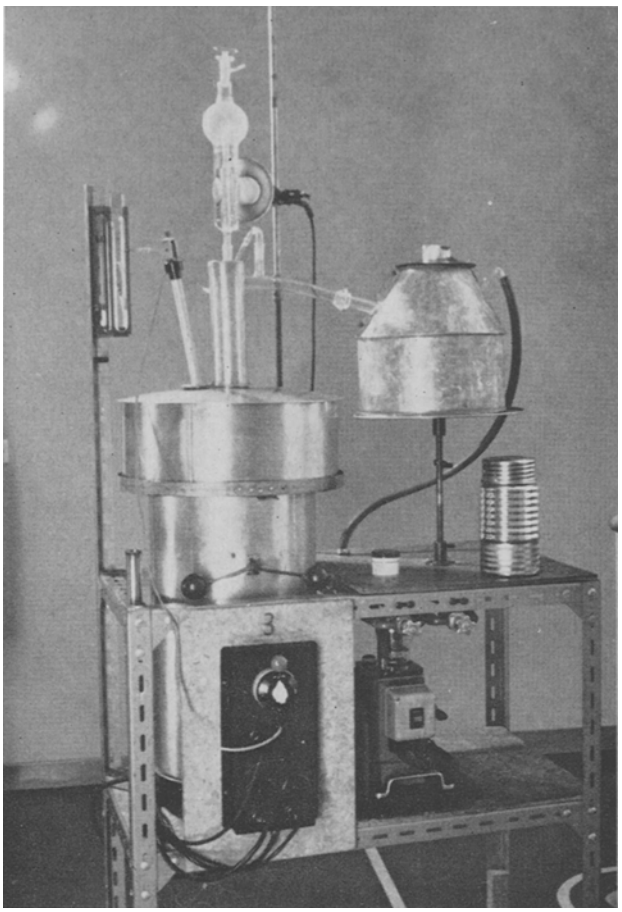

FIG. 2. Deodorizer

60C. During the heating, and at the beginning of the deodorization, some non-condensable volatiles are given off, but after about 10 min of deodorization at 180C the vacuum pump can be stopped. As a control of tightness we generally finish the deodorization without using the vacuum pump at all. Before the oil is cooled, the radiation heater at the steam generator is turned off to avoid wet oil. In a series of deodorized coconut oils we found less than 0.02% of water present.

The ground glass joints are greased with Dow Silicone high vacuum grease. For the neck joint on the deodorizer we have tried the Teflon sleeves, but they were not always tight. Despite limiting the application of the grease to the upper third of the joint and applying the smallest amount possible the Silicone grease may be squeezed out on the inside where it contacts splashes of hot oil. Therefore, A29 neck joints (45 mm length) would now be preferred.

We tried to deodorize 1000 g of soybean oil without

TABLE I

Taste versus rate of steam and time of deodorization

Rate steam flow		15 g H_2O per h						30 g H_2O per h					
Minutes at 180C		60		120		240		30		60		120	
Total % steam		1.5		3.0		6.0		1.5		3.0		6.0	
Storage	Series	T[a]	R[b]	T	R	T	R	T	R	T	R	T	R
0	1	6.2	11	6.5	11	6.3	14	6.7	9	6.5	13	6.2	14
	2	6.2	16	6.5	12	6.7	8	6.0	13	6.2	13	6.2	10
3 days at 50C	1	5.8	11	5.8	12	5.8	13	5.5	14	5.5	14	6.0	8
	2	4.5	16	5.0	12	5.2	8	5.3	14	5.2	12	5.2	10

[a] T = taste as expressed on a 10 point scoring scale.
[b] R = sum of ranks; to obtain average rank divide by 6.

TABLE II

Taste versus deodorization temperature

Rate of steam		30 g H_2O per h									
Minutes at 180C or above		30				120					
Total % of steam		1.5				6					
Maximal temp.		180		210		240		180		210		240	
Min at max temp		30		24		15		120		114		105	
Storage	Series	T[a]	R[b]	T	R	T	R	T	R	T	R	T	R
0	1	7.0	13	6.7	13	6.8	10	6.5	14	7.2	8	6.2	14
	2	6.2	15	6.3	8	6.2	13	5.2	17	6.0	7	5.8	12
3 days at 50C	1	5.3	14	5.5	13	5.5	9	5.8	9	5.3	11	5.2	16
	2	5.5	13	5.7	11	5.5	12	5.3	13	5.5	7	4.8	16

[a, b] See Table I.

and with about 1 g of the silicone grease. There was no effect on the taste.

Taste evaluations of deodorized oils

A few experimental data are presented in order to give an idea of the reproducibility of the apparatus.

Taste tests. Groups of 3 oils were tasted by 6 selected tasters. Two such groups were tasted at one session. Within each group the 3 oils were ranked 1, 2, and 3, 1 being the most neutral oil. The ranks were summed for the 6 tasters and the sum of ranks were analyzed statistically. The tasters were also asked to score the oils on a 10 point scoring scale, where 8 signifies a practically neutral oil and 4 an oil with a pronounced taste. Certain deodorized oils obtain average initial scores of 8, while soybean oils generally obtain 6 or 7, depending on the sensitivity of the tasters.

Experiment

Large (1000 g) deodorizations were carried out using only one lot of alkali refined and bleached soybean oil.

The time of deodorization was varied at each of two rates of steam flow (Table I). No significant differences were found. At the lowest steam rate the taste of the oil with 1.5% steam seemed low, but this may be due to chance. Whether we deodorize at 15 or 30 g of water per hr depends on the amount of splashing which can be allowed.

The temperature of deodorization was varied in 30 and in 120 min-runs, the time being calculated from the moment the temperature of the oil had reached 180C.

The 210C-oils were preferred to the 180 and 240C-oils in a slight majority of the comparisons. Analysis of variance on the sum of ranks shows that the difference is significant on the 1% level. It seems (level of significance 5%) that the difference is more pronounced in the 120 min-runs than in the 30 min-runs but 30 min of deodorization seems to be sufficient.

Only one lot of soybean oil was used, i.e. the author does not know if the conclusions would be the same for other batches of raw material.

ACKNOWLEDGMENTS

Assistance in development of the apparatus by J. Gravenhorst; statistical analysis by G. E. Hjort; and permission for publication by Aarhus Oliefabrik A/S.

REFERENCES

1. First factory operation on record in Aarhus is June 1896.
2. Schwab, A. W., and H. J. Dutton. JAOCS *25*, 57 (1948).

[Received July 23, 1962—Accepted January 28, 1963]