

Laboratory deodorization: An overview of past and present equipment and practical uses

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Process changes in any stage of the refining of edible oil can have dramatic effects on finished oil quality. Shifts in consumer preferences for attributes of edible oils are forcing refineries to re-evaluate processing protocols and equipment as well as to use more active bleaching earths. Many research and development edible oil chemists are evaluating the effects these changes may have on finished oil quality.

Although deodorization is critical to the evaluation of finished oil quality, many projects stop short of the deodorization step because equipment, materials, and time are unavailable or costly.

This paper presents laboratory deodorization options, defines laboratory-scale deodorization, describes its practical uses, and provides an overview of past and present deodorization techniques and equipment. In addition, data are presented from a newly designed six-port microdeodorizer.

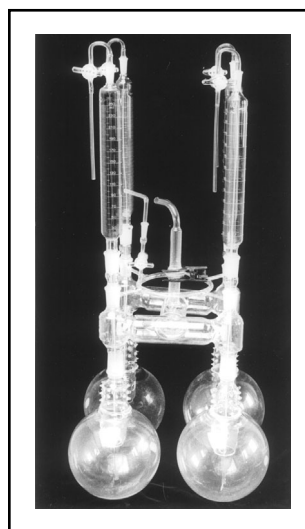


Figure 1. Schwab and Dutton deodorization equipment modified by Moulton.

DEODORIZATION PRINCIPLES

The theory of edible oil deodorization was first described by A.E. Bailey in 1941 and has been presented in a number of reviews, most recently by S.C. Loft in 1990. Deodorization, in principle, is governed by basic

laws of distillation involving Dalton's law of partial pressure and Raoult's law governing liquid and vapor phase equilibria. Variables affecting deodorization include temperature, time, pressure (vacuum), agitation, and stripping steam. Steam or an inert gas is employed as a carrier to increase agitation, break down fatty hydroperoxides, and distill volatile components from the nonvolatile triglyceride oil.

TYPES OF DEODORIZERS

Commercial steam deodorizers can be batch, semicontinuous, or continuous, but batch type deodorizers are the most commonly used for laboratory work. Continuous laboratory deodorizers were described by Bitner *et al.* in 1986 and Smouse in 1997. Batch deodorizers bubble steam through heated oil for longer periods of time (1–3). In continuous deodorizers, oil passes countercurrent to the steam as it flows through a series of distillation trays. Residence time in a commercial continuous deodorizer varies from 20 to 30 min. Although continuous deodorizers are more applicable than batch to current practices, there are very few continuous laboratory deodorizers in use today.

Hidden behind this paradox between the processes, commercial and laboratory batch, semicontinuous, or continuous deodorizers is the inherent problem of comparing data. Batch deodorizers tend to be used when comparisons can be based on relative numbers. Continuous deodorizers

Figure 2.

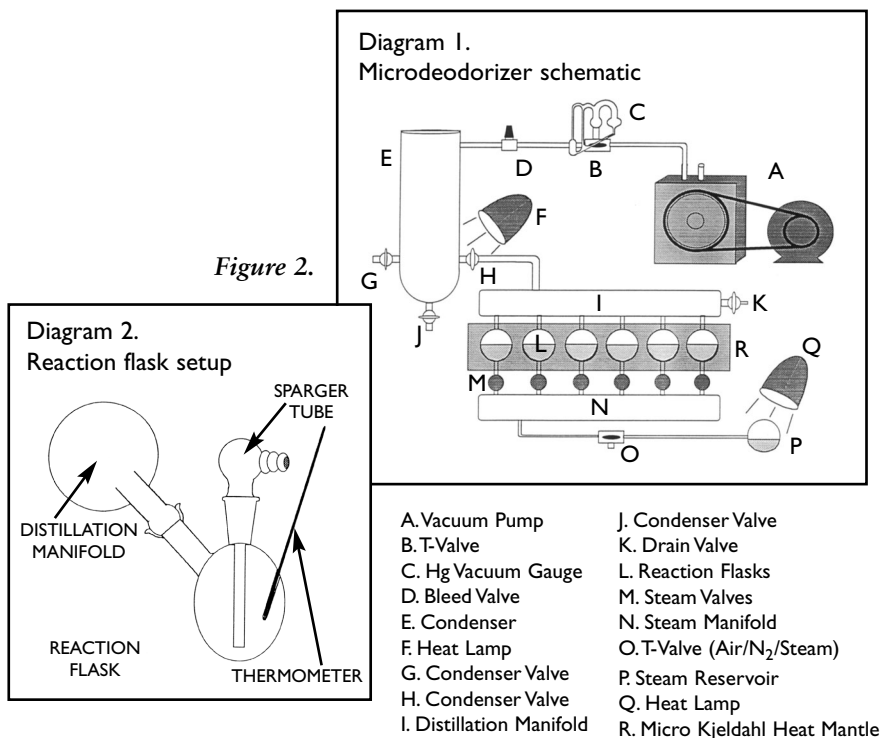


Table 1. Typical conditions for batch vs. continuous laboratory deodorizers

Process stage		Batch	Continuous/ semicontinuous
Deaeration	Time	<30 min	<30 min
	Temperature	60–100°C	60–100°C
	Vacuum	<1 mm Hg	<1 mm Hg
Steam stripping	Time	1–4 hr	3–9 min
	Temperature	250–275°C	200–260°C
	Vacuum	<1 mm Hg	<1 mm Hg
	Steam rate	3–10%	1.3–12.1%
Holding	Time	<1 hr	<30 sec
	Temperature	<100°C	<60°C
	Vacuum	<1 mm Hg	<1 mm Hg
Cool-down	Filter	Yes	No reference
	Sparge	N ₂	N ₂
	Freeze	Yes	Yes

are usually employed when more nearly absolute numbers are needed. Smouse presented many of the various designs of continuous and semicontinuous laboratory deodorizers. The cost estimates of such designs range between \$10,000 and \$15,000. Batch deodorizers tend to be less costly and can be fabricated from more readily available equipment than continuous systems. The simplest design of a batch-type laboratory deodorizer was described by Mounts and colleagues in 1969 at an estimated price of \$3,000 to \$4,000 and consisted of a high-vacuum pump, Dewar condenser, three-necked distillation flask, thermometer, manometer, and heating mantle.

Descriptors for laboratory deodorizers include *laboratory*, *micro*, *mini*, *bench*, and *smallscale*. It is confusing when the capacities of such equipment range from gram quantities up to 50 kg. For clarity, we suggest that a laboratory deodorizer be defined as one whose capacity ranges from 60 to 5000 g. As a further dividing line, we suggest that the term *micro* apply to deodorizers with <200-g capacity and *common* apply to deodorizers with 200 to 5000-g capacity. This separation by capacity will differentiate between deodorizers that will and will not have oil capacity to produce enough oil for organoleptic evaluations. Generally speaking, sensory testing of initial quality and after accelerated storage requires about 300 g of oil.

workers utilize silicone grease for high-vacuum work, deodorizer joints lubricated with silicone grease can contaminate the oil. In such circumstances, the deodorized oil may become cloudy and exhibit elevated silicone levels. When using silicone grease, the best practice is to use as little grease as possible at joints coming into contact with hot oil over prolonged periods of time or to use a nonsilicone grease such as vegetable oil-based grease or a high molecular weight/polymer-based grease, e.g., Celvacene or Lubriseal (Fischer Scientific, Itasca, IL).

The nature of the coolant used in the condenser is important to the maintenance and care of the vacuum pumps used in the system. Deodorizer distillates contain water and many products that can affect the viscosity and performance of the vacuum pump oil. Many of the commonly used salt and ice slurries do not have the cooling capacity to condense the low molecular weight contaminants. Acetone with dry ice is one of the more common coolants used.

COMMON APPLICATIONS

Selection of laboratory deodorizers should be based on four variables: degree of confidence in or reproducibility of data, type of data required (relative compared with absolute), capacity needed to complete the analytical workup, and cost or availability of equipment.

The underlying objective in any oil-pro-

TYPICAL CONDITIONS

Deodorization in simple terms is a high-temperature, high-vacuum, steam distillation of low molecular weight volatiles from nonvolatile triglycerides. The process, batch or continuous, can be broken down into four stages: de-aeration, steam stripping, holding, and cool-down. Typical conditions for laboratory deodorization are listed in Table 1.

Although many

cessing project is to establish a standardized means to compare results between data sets. Applications for laboratory deodorizers, whether based on relative or absolute numbers, must be proven reproducible.

A majority of all deodorization work can be performed using a batch deodorizer. Relative effects of process changes upstream of the deodorization stage, such as clay dosage, clay types, water wash compared with no water wash, etc., can be determined using batch deodorization. Process effects of deodorizer conditions on a given oil require the type of absolute numbers that only a continuous deodorizer can provide.

Capacity and cost are two critical issues. Advances in instrumentation and micro techniques have dramatically reduced the amount of oil needed to evaluate quality. Today, analyses for color, chlorophyll, peroxide value (PV), free fatty acid (FFA), oxidative stability index (OSI), fatty acid composition by gas chromatography (GC), and volatiles can all be performed on a minimum of 60 g of oil.

The cost of laboratory deodorizers is directly proportional to the capacity needed. In most studies, batch-type deodorizers are more commonly used because the capacities needed fall within that of common glassware and heating units. Batch deodorizers also tend to cost less than continuous deodorizers. A simple batch deodorizer setup with 100- to 500-g capacity will cost about \$4000 per unit. Single 11-L units described by Moulton also approach \$4000 per unit. The cost of a single continuous deodorizer unit approaches \$10,000 to \$15,000.

One other aspect of continuous units is that cleaning protocols can be time-consuming due to the complexity of the equipment. Batch deodorizers tend to have simpler parts that can be interchangeable and purchased in quantities that allow for multiple instead of single runs per day.

STATISTICAL APPLICATIONS

Schwab and Dutton published one of the most-cited regarding laboratory deodorizers (Figure 1), which offers a standardized method to deodorize four independent

samples simultaneously under identical conditions of time, temperature, and steam stripping.

Currently, with the evolution of statistics and multiple-variable experimental designs, there is a demand to deodorize more samples in shorter times. To meet this demand, we introduce a modified micro-sized version of the Schwab deodorizer (Figure 2). Advantages of this design are that it provides simultaneous deodorization of six 75–10-g, independent samples in three to four hours.

Table 2. Comparison of color data across each port of the new deodorizer

RBD red color ^a						
Sample #	Port 1	Port 2	Port 3	Port 4	Port 5	Port 6
1	1.0	1.0	1.1	1.1	1.0	1.1
2	1.0	0.9	0.9	0.9	1.0	1.0
3	2.3	2.2	2.3	2.3	2.3	2.3
4	0.8	0.9	1.0	0.9	1.0	0.8

RBD yellow color ^b						
Sample #	Port 1	Port 2	Port 3	Port 4	Port 5	Port 6
1	6.9	6.6	7.3	7.3	6.8	6.8
2	8.4	7.9	8.3	8.3	8.6	8.4
3	14.0	13.0	13.0	13.0	14.0	13.0
4	13.0	13.0	13.0	13.0	13.0	13.0

^a Analysis of variance (ANOVA) of refined, bleached, deodorized (RBD) red color $F_{cal} < F_{crit}$, $\alpha = 0.5$ ($0.0118 < 2.773$).

^b ANOVA of RBD yellow color $F_{cal} < F_{crit}$, $\alpha = 0.5$ ($0.0077 < 2.773$).

The basic design of the microdeodorizer consists of the following: steam generator, steam manifold, reaction flask, six-port heater, distillation manifold, condenser, vacuum gauge, and high-vacuum pump (Fig. 2, Diagrams 1 and 2).

The microdeodorizer was assembled for \$6,500. Fifty percent of the cost went for customizing the glassware (Universal Instruments, Palatine, Illinois). The remainder of the cost was distributed between the Micro Kjeldahl Heater (Labconco, Kansas City, MO), the Welch Dual Seal model 1400 vacuum pump (Fisher Scientific), and miscellaneous supplies (e.g., thermometers, highvacuum tubing, clamps, etc.).

The following protocol can be used. A

sample (75–100g) of refined bleached oil is transferred to the reaction flask. The flask is mounted on the heater and connected to the steam and distillation manifolds. The sample is de-aerated for 15 min. under vacuum (<1 mm Hg). The sample is then heated and sparged with steam for 1–3 hours at temperatures of 240–270°C. The sample is then cooled under vacuum. Steam is shut off when the sample reaches 100°C. Cooling is continued until the oil temperature reaches 60°C. Vacuum is then broken with nitrogen. Each sample is then filtered through fluted

filter paper (Fisher-brand P8 Qualitative, Coarse, Fast Flow; Fisher Scientific) and nitrogen-sparged before being sealed in a bottle and stored in a freezer (–5°C).

Performance evaluations were examined in a series of tests.

TEST ONE

Four bleached soybean oil samples were deodorized at each port under the following standard set of conditions: vacuum, <1mm Hg; steam rate, 5%; 260°C; and residence time, 1

hour. Analysis of variance of the Lovibond red and yellow color values (AOCS Official Method Cc 13b-45, Automated Lovibond Tintometer PFX990) showed that there was no difference between oils generated in each port (Table 2).

TEST TWO

Refined canola oil was treated/bleached under multiple conditions followed by subsequent deodorization in duplicates. Port positions and selection of samples were randomized. Conditions were the same as Test 1. Pooled data from 95 pairs of data sets resulted in a standard deviation of 0.09 for red colors (range 0.4 to 3.5) and 1.83 for yellow colors (range 2.3 to 70).

TEST THREE

Standardization in the case of both the microdeodorizer and the Schwab deodorizer applies more to the step-by-step protocol than to creation of absolute values. In practice these deodorizers are used more for generating relative values than absolute ones. This is evident when industrial and laboratory deodorized oil results under a standard set of conditions are compared. Matched sets of bleached and deodorized oil samples from various producers were deodorized in the laboratory under the same conditions as Test 1. Color, FFA, and PV analyses using Official AOCS Methods are reported in Table 3.

It would be desirable to standardize deodorization conditions to be applicable to all oils. However, variations in feed stock quality, industrial equipment, and process conditions as well as differences due to the nature of each given oil make it impractical to do so. A comparison of laboratory and plant data, however, shows good correlation between the two data sets. Closer scrutiny of the data shows that the base range is probably too broad. Inspection of each individual oil group shows that, as expected, some diversity exists between laboratory and plant data. In general, the plant data had higher PV and FFA values than the laboratory data. This can be attributed to the fact that the deodorized oil samples from the plants were analyzed after 2 to 20 days after shipping and handling. Commercial oil will also contain up to 50 ppm citric acid, which will affect the final FFA content.

Corn oil showed the most variation between laboratory and plant data. In circumstances such as these, one would have to set up an experimental matrix to establish the best set of conditions to attempt to make absolute comparisons between plant and laboratory data.

A simple test was conducted to demonstrate the diversity of treating the bleached corn oil samples from the different producers. Wet-milled corn oil samples from producers I and K and dry-milled corn oil from producer L were subjected to a simple experiment where deodorization time and tem-

Table 3. Data on deodorization in a plant vs. laboratory

Oil Type	Producer	Deod	RBD data												
			Red			Yellow			Chlorophyll	% FFA			PV		
		Temp (°C)	Lab scale	Plant scale	Δ	Lab scale	Plant scale	Δ			Lab scale	Plant scale	Δ	Lab scale	Plant scale
Palm	A	260	4.4	4.5	-0.1	70	70	0	0	0.038	0.120	-0.082	0	0.2	-0.2
	A	260	3.3	3.1	0.2	50	50	0	0	0.018	0.080	-0.062	0	0.1	0.1
	A	260	3	3.2	-0.2	50	38	12	0	0.032	0.100	-0.068	0	0.05	-0.05
	B	260	2.9	2.3	0.6	43.5	24	19.5	0	0.026	0.100	-0.074	0.05	0.15	-0.1
	B	260	2.9	2.4	0.5	42.5	28	14.5	0	0.080	0.100	-0.020	0	0.15	-0.15
	B	260	3.25	2.4	0.85	50	24	26	0	0.100	0.120	-0.020	0	0.1	-0.1
	C	260	2.9	3	-0.1	50	50	0	0	0.000	0.007	-0.007	0	0.1	-0.1
Soybean	D	260	0.85	0.8	0.05	6.3	4.7	1.6	26	0.023	0.023	0.000	0	0.05	-0.05
	E	260	0.4	0.6	-0.2	2.7	5.2	-2.5	21	0.025	0.025	0.000	0	0.05	-0.05
	F	260	0.4	0.5	-0.1	2.35	5.4	-3.05	16	0.028	0.031	-0.003	0	0	0
	E	260	0.4	0.4	0	2.7	2.7	0	28	0.011	0.010	0.001	0.05	0	0.05
	G	260	1.1	0.8	0.3	5.5	5.3	0.2	34	0.010	0.023	-0.013	0	0	0
Canola	H	260	0.8	0.7	0.1	5.6	5.4	0.2	19	0.010	0.010	0.000	0	0	0
	H	260	0.85	0.7	0.15	6.55	5.3	1.25	21	0.021	0.015	0.006	0	0.05	-0.05
	H	260	0.75	0.7	0.05	5.4	5.5	-0.1	25	0.018	0.020	-0.002	0	0	0
	H	260	0.9	0.8	0.1	6.8	5.7	1.1	24	0.010	0.010	0.000	0	0	0
	H	260	0.75	0.8	-0.05	5	5.3	-0.3	11	0.005	0.010	-0.005	0	0.05	-0.05
	H	260	0.75	0.9	-0.15	5.6	6.4	-0.8	19	0.010	0.005	0.005	0	0	0
	H	260	0.9	1	-0.1	6.8	9.1	-2.3	24	0.031	0.020	0.011	0.05	0.05	0
Peanut	D	260	0.6	1	-0.4	3.2	4.55	-1.35	0	0.310	0.440	-0.130	0	0	0
Rice	D	260	3.45	3.2	0.25	30	32	-2	0	0.064	0.038	0.026	0	0	0
Corn	I	260	1.1	1.5	-0.4	6.9	9.9	-3	0	0.331	0.054	0.277	0	0.05	-0.05
	I	260	1.2	1.4	-0.2	7.6	9.7	-2.1	0	0.441	0.059	0.382	0.05	0.05	0
	I	260	1.2	1.6	-0.4	7.6	12	-4.4	0	0.345	0.050	0.295	0	0	0
	I	260	1.2	1.7	-0.5	8.8	12	-3.2	0	0.204	0.056	0.148	0	0.05	-0.05
	K	260	2.1	2.5	-0.4	15	26	-11	0	0.113	0.073	0.040	0	0.21	-0.21
	L	260	3.5	2.4	1.1	26	17	9	0	0.298	0.169	0.129	0	0	0

Deodorization time, vacuum, and temperature the same: 1 hr at ~1.0 mm Hg and 260°C

Table 4. Comparison of RBD corn oil data

Producer	Process	Conditions		FFA		OSI		Color red/yellow		
		Deodorizer	°C	1 hr	2 hr	1 hr	2 hr	1 hr	2 hr	
I	Wet	Laboratory	250	—	—	—	—	—	—	—
			260	0.441	—	3.00	—	1.2 / 7.6	—	
			270	0.062	—	5.75	—	1.2 / 6.0	—	
K	Dry	Plant	250	0.059	6.10	—	1.4 / 9.7	—	—	
			Laboratory	250	—	0.122	—	5.18	—	2.1 / 13
			260	0.113	—	5.30	—	2.1 / 15	—	
L	Wet	Plant	270	0.078	—	5.45	—	2.0 / 12	—	
			Laboratory	250	—	1.63	—	1.73	—	3.5 / 28
			260	0.298	—	3.45	—	3.5 / 26	—	
L	Plant	Plant	270	0.331	0.160	2.93	3.43	3.4 / 26	2.9 / 21	
			260	0.331	0.160	3.95	—	2.4 / 17	—	

perature were varied. The results are shown in Table 4. Color and FFA levels agreed with the values from the plant sample after one hour at 260°C. The wet-milled samples required higher temperatures and longer times to compare with the plant samples.

The practical value of the microdeodorizer is apparent when comparing relative numbers. For example, Table 5 lists the relative data from a plant trial in which various bleaching clays were used to treat canola oils. The OSI of the RBD oils from the plant were compared to the data from laboratory deodorized samples using the bleached oil samples from the plant. Although there is a definite difference between the absolute numbers, the relative order of stability was maintained from plant to laboratory.

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Table 5
Rank data of laboratory vs. plant OSI data

Sample	Laboratory OSI 1	Laboratory OSI 2	Average OSI	Rank order (1 = best)
1	7.85	7.60	7.73	3
2	6.70	7.25	6.98	4
3	8.15	8.45	8.3	2
4	8.55	8.55	8.55	1
5	6.10	6.85	6.3	5

Sample	Plant OSI 1	Plant OSI 2	Average OSI	Rank order (1 = best)
1	8.10	7.95	8.03	3
2	7.50	7.15	7.33	4
3	8.55	8.75	8.65	2
4	8.95	8.85	8.90	1
5	7.30	6.40	6.85	5

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