Effects of Deodorization and Steam-Refining Parameters on Finished Oil Quality

A. Maza*, R.A. Ormsbee and L.R. Strecker
Best Foods Research and Engineering Center CPC International, Inc., Union, New Jersey 07083

Deodorization and steam-refining parameters have significant impact on the quality of finished oil. Hence, optimization of parameters is critical for the production of acceptable oil. Temperature, time, throughput rate, sparging steam rate and pressure must be optimized to produce edible oil with desirable characteristics. Process optimization modeling in the laboratory, pilot plant and refinery is utilized to obtain predictive equations correlating product characteristics with deodorization parameters and feedstock properties. This optimization technique is demonstrated on model feedstocks and typical commercial oil. Finished corn and soybean oil quality are expressed by tocopherols and free fatty acid levels and sensory attributes during accelerated storage.

KEY WORDS: Corn oil, deodorization, flavor, optimization, quality, response surface, soybean oil, stability, steam refining, tocopherol.

Deodorization and steam-refining/deodorization processes employ the principles of steam distillation for a complex matrix of vegetable oils. Within this matrix, there is a marked difference in molecular weight and, consequently, volatility of triglycerides, tocopherols, sterols, free fatty acids, pesticides and oxidative intermediates representing the majority of odoriferous and strongly flavored compounds. Although steam distillation targets only the undesirable compounds, a simultaneous loss of valuable oil components (*e.g.*, triglycerides, natural antioxidants) is unavoidable.

During steam stripping, complex chemical and physical phenomena are taking place. The chemistry involved includes thermal decomposition of oxidation products and pigments, hydration of conjugated polyenic compounds and hydrolysis of triglycerides generating free fatty acids. The main physical effects are vaporization of volatiles and the entrainment of neutral oil droplets in the stripping steam. Both hydrolysis and mechanical entrainment of neutral oil are responsible for the substantial yield decrease during deodorization, while excessive removal of natural antioxidants leads to reduction of finished oil stability. Theoretical equations, defining the effect of steam stripping, apply strictly to ideal solutions of foreign materials in the oil (1).

The vegetable oil matrix, however, is a nonideal solution, and the vaporization efficiency of volatile compounds strongly depends on oil composition, deodorization parameters, equipment type and condition, and operating procedures. Thus, precise theoretical calculation of optimum deodorization parameters is difficult and results in predictions that significantly deviate from practice. Since finished oil quality [absence of pesticides, low free fatty acid (FFA), acceptable flavor and stability] depends on a large number of deodorization or steam-refining variables and interactions among them, process optimization based on statistical design and interpretation of data provides the best option.

The scope of this paper includes the results generated through statistically designed experiments conducted on bench, pilot plant and plant scale with different sets of variable parameters and oil types. The experimental results were evaluated through regression analysis to produce mathematical models and response surfaces for selected finished-oil quality indicators (*e.g.*, tocopherol retention, color, residual FFA, flavor). The evaluation of response surfaces defined the optimum conditions and, by contrast, the unacceptable domains for each scale of processing. The studies indicated conclusively that oil quality is delineated as a complex result of overlapping responses, which need therefore to be judges in context.

*To whom correspondence should be addressed at Best Foods Research and Engineering Center, 1160 Commerce Ave., Union, NJ 07083.

EXPERIMENTAL PROCEDURES

Laboratory-batch steam refining/deodorization. The experiments were conducted in 2,000-g batches at 0.1–0.5 mm Hg absolute pressure for 2 h. The equipment was a custom-made glass deodorizer, consisting of a one-piece 5-L flask equipped with an upper demister baffle, a built-in sparging steam inlet, a thermometer well and a 2.5-inch diameter vapor duct. The one-piece construction eliminated the exposure of joints to high temperatures, thus preventing air leaks during deodorization. The deodorizer was connected through a hot well to dry-ice traps and further to a vacuum pump. Stripping steam was produced from distilled, deaerated water, metered and evaporated in a glass steam generator connected directly to the deodorizer steam inlet. Both the steam generator and deodorizer were heated in temperature-controlled electrical mantles.

Pilot-plant batch deodorization. Steam refining/deodorization was conducted in a batch, 90/lb nominal capacity, SS 306 deodorizer, constructed by Wurster & Sanger Inc., Chicago, IL, equipped with a 3 × 6 kW external zone electrical heating system and serviced by a three-stage steam ejector vacuum system with inter-stage condenser providing 1–2 mm Hg absolute pressure.

For each experiment, the deodorizer was filled with 90 lb (40.9 kg) of feed oil, which was then preheated at a gradient of 12°F (7°C)/min from 80 to 250°F (27–121°C); minimal agitation was provided with nitrogen to ensure satisfactory heat transfer. Subsequently, the batch was heated from 250°F (121°C) to the test temperature at a gradient of 6°F (3°C)/min and steam (1%/h rate) was substituted for nitrogen. At the design temperature, the rate of sparging steam was adjusted according to design requirements. Each batch was sampled at 30-min intervals by withdrawing oil under vacuum without interrupting the process. Actual steam refining/deodorization lasted for 150 min, after which the batch was cooled by a procedure that was the reverse of heating. All deodorized oils were treated with an aqueous solution of citric acid (equivalent to 50 ppm anhydrous) during the cooling stage.

Plant continuous deodorization. Plant-scale deodorization was conducted in a continuous mode in a SS 304 model "C" deodorizer, designed and constructed by EMI (Des Plaines, IL). Typical alkali-refined and bleached soy-

bean oil, produced from Delmarva crude stock and accumulated in one tank, was used as feed for the entire experiment. As a result of the constant FFA level in this feedstock and the same stripping steam rate, the deodorizer vacuum also remained constant throughout the six experiments.

Each test point was run for 4 h of steady-state operation. At time = 0, the design deodorization temperature and oil rate were set by means of the deodorizer controls. At time = 2 h, the deodorizer was judged to have reached equilibrium and to be producing finished oil representative of test variables. Deodorized oil samples were taken at 4, 5 and 6 h (equivalent to 2, 3 and 4 h of steady-state operation, respectively). Following the last sampling, the conditions were adjusted for the next test point.

Analytical methods. FFA and peroxide value (PV) analyses were performed by AOCS Official Methods Ca 5a-40 and Cd 8-53, respectively (2). Color, anisidine value (AV), tocopherols and pesticide residues were determined by procedures described in our previous publications (3,4). Volatile compounds (GCV) were obtained by purge-and-trap gas chromatography with a Hewlett-Packard 7675 A sampler (Palo Alto, CA), interfaced with a gas chromatograph equipped with a 12 foot × 1/8 inch diameter stainless-steel column, packed with 10% Carbowax 20M-TPA, 80–100 mesh on Chromosorb WAW (Supelco Inc., Bellefonte, PA) and a flame-ionization detector.

Oil evaluation. Oil aging was simulated through one or more storage tests: (i) color reversion [stored at 75°F (24°C) in loosely capped clear glass bottles, without exposure to light], (ii) dark storage [stored at 95°F (35°C) with 10% air headspace in tightly capped clear glass bottles, without exposure to light], (iii) light storage [stored at 95°F (35°C) with 10% air headspace in tightly capped clear glass bottles, continuous exposure to 50 foot-candle light].

Sensory evaluation of fresh as well as aged oil samples was conducted. Samples were prepared for flavor evaluation and were presented to trained panelists following the procedure outlined in Method Cg 2-83 (2). Panelists rated the oils for flavor strength by an internal method similar to the AOCS Intensity Scale. This internal procedure used a flavor strength scale, ranking oils from 1 (bland) to 9 (extremely strong), as opposed to the AOCS scale that ranks oils from 10 (bland) to 1 (extreme). The Best Foods method combines the AOCS intensities of 9 (trace) and 8 (faint) into ranking 2 (almost bland).

RESULTS AND DISCUSSION

The determination of optimum deodorization and steam-refining/deodorization parameters is critical not only to producing good-quality oil, but also to minimizing losses resulting from hydrolysis at high temperatures or entrainment due to high sparging steam rates. Thus, a statistical experimental approach provides the means for reliable assessment of each deodorization system to achieve desirable oil quality while alleviating the negative effects of possible under- or over-stripping.

Laboratory-batch steam refining/deodorization. The objective of the steam-refining/deodorization study was to determine the effect of temperature, sparging steam rate and their interaction on finished-oil quality indicators and to define the acceptable ranges for bench processing. A

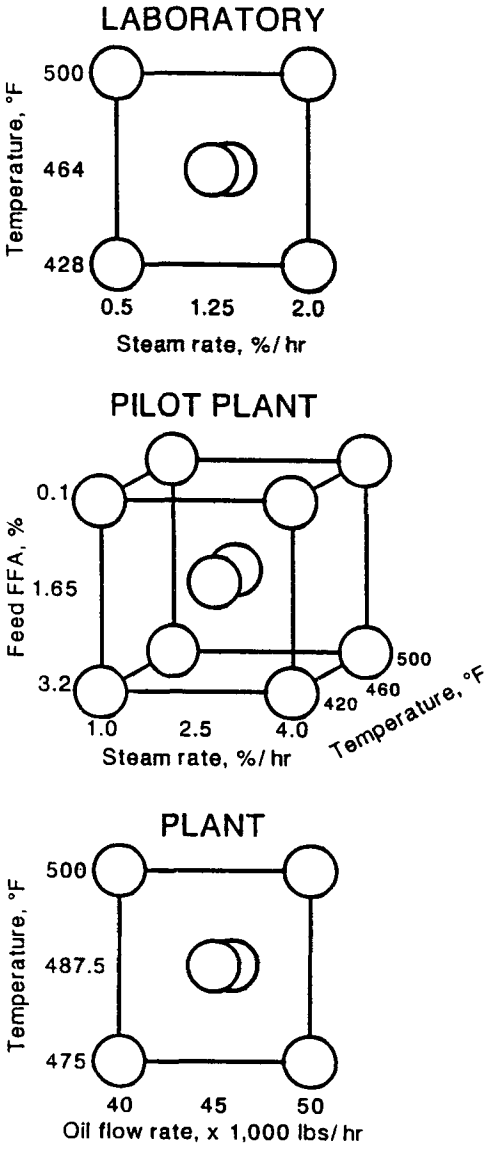

FIG. 1. Deodorization and steam-refining—experimental designs.

2^2 factorial design with duplicated centerpoint consisting of six random-order experiments, of which four contained combinations of high/low temperature/steam rate, was employed for the laboratory optimization (Fig. 1). For any of the finished-oil quality indicators (Y) evaluated, a mathematical model was developed that indicates the linear effect of variables selected (X_1, X_2) or their interactions (X_1X_2) according to this equation:

$$Y = A_0 + A_1X_1 + A_2X_2 + A_3X_1X_2 \quad [1]$$

The experiments covered a temperature range of 428–500°F (220–260°C) and a range of sparging steam rates of 0.5–2.0%/h. To clearly differentiate the process responses, the ranges were selected so that both good- and poor-quality finished oils were produced.

The feedstock was typical bleached corn oil derived from crude recovered directly from wet-milled corn germ by a patented process (5). The bleached oil exhibited light color

TABLE 1

Laboratory[a] Steam Refined/Deodorized Corn Oil

Variable parameters					
Temperature (°F)	428	428	464	500	500
Steam rate (%/h)	0.5	2.0	1.25	0.5	2.0
Oil characteristics[b]					
FFA (%)	0.105	0.025	0.012	0.020	0.010
Color (Y/R)					
0 wk	3/0.4	2/0.1	3/0.3	2/0.2	2/0.2
8 wk	17/1.4	12/0.7	16/1.2	14/0.9	19/1.2
Peroxide value (meq/kg)					
0 wk	0	0	0	0	0
8 wk	3.9	1.1	1.6	1.9	3.0
Tocopherols (%)					
0 wk	0.160	0.124	0.095	0.118	0.024

[a]Constant parameters: 2,000 g oil (1.7R in 5.25-inch cell, 1.24% FFA, 0.160% tocopherol), 0.1-0.5 mmHg absolute pressure, 2 h.
[b]Color reversion test (75°C, loosely capped glass bottles, dark storage). FFA, free fatty acid.

(20Y/1.7R), moderate FFA (1.24%) and a high tocopherol content (0.160%).

Table 1 shows the characteristics of finished oils produced initially and during the color reversion test. Since the replicate centerpoint samples exhibited identical initial qualities, only one was subjected to stability evaluation. The low-temperature/low-steam rate experiment [428°F (220°C) & 0.5%/h] resulted in understripping (unsatisfactory FFA removal and no tocopherol loss), while at the other extreme, the high temperature/high steam rate experiment [500°F (260°C) & 2.0%/h] produced an overstripped oil (containing the lowest FFA and only 15% of the original tocopherol content). The oils from these two extreme experiments were more susceptible to color reversion and oxidation compared to the remaining samples. The accelerated storage tests revealed stronger initial and over-time flavors in samples deodorized with low sparging steam rates (0.5%/h) as shown in Figure 2.

To facilitate proper statistical interpretation of the data, the requirements for a good-quality finished oil were established as follows: FFA 0.02% maximum, tocopherol retention (TOC) = 50% minimum (equivalent to 0.080% absolute tocopherol content), initial flavor strength (F_o) = 2.0 maximum, flavor strength after 4 wk of storage with light exposure (F_1) = 4.0 maximum and flavor strength after 2 mon of storage in dark (F_2) = 3.6 maximum. Regression analysis performed on these oil quality indicators produced the following predictive equations but did not establish an acceptable model for residual FFA:

		R^2	Adj. R^2	
TOC, %	= 97.186 + 182.361s − 0.451ts	0.953	0.922	[2]
F_0	= 2.453 − 0.0008ts	0.851	0.801	[3]
F_1	= 15.976 − 0.023t − 5.826s + 0.010ts	0.977	0.902	[4]
F_2	= 13.4372 − 0.019t − 7.144s + 0.014ts	0.997	0.989	[5]

where t = temperature (°F); s = sparging steam rate (%/h).

Response surfaces presented in Figure 3 indicate a shaded region where all the above requirements for an acceptable finished oil are met.

Because both samples steam refined/deodorized at 428°F (220°C) showed either higher than desirable acidity

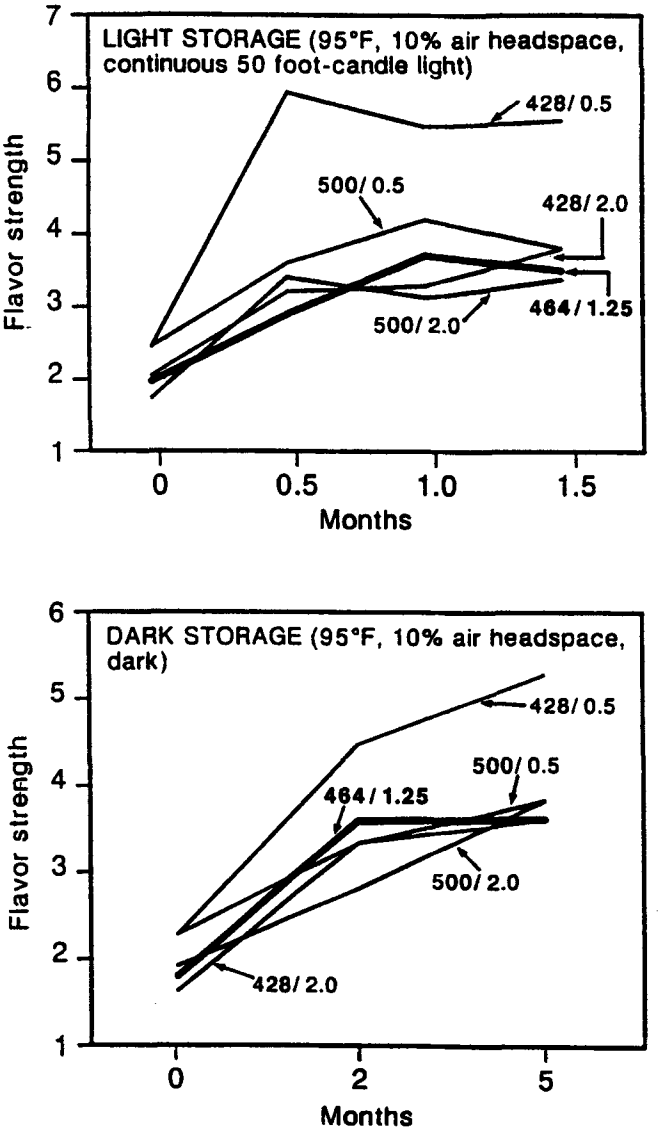

FIG. 2. Flavor stability of laboratory steam refined/deodorized corn oil.

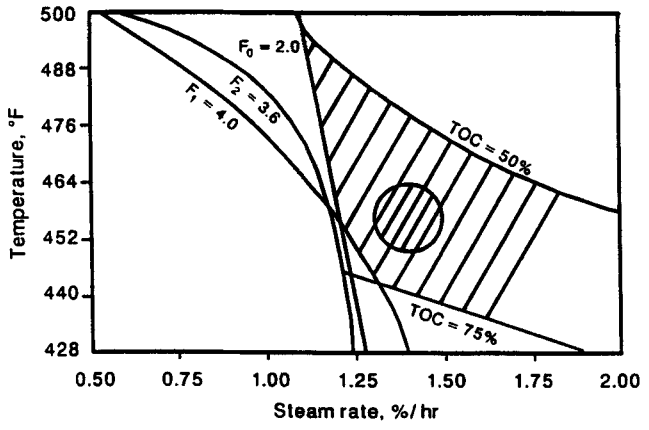

FIG. 3. Laboratory steam refining/deodorization optimization—oil-quality response surfaces.

or poor flavor stability, the low temperature tested is unsatisfactory. The optimum parameters appear to be within the shaded area, and especially around the darkly-shaded circle, corresponding to 450–460°F (232–238°C) and 1.35–1.45%/h sparging steam rate. Processing at the optimum parameters produces oils with the best initial characteristics and stability and is therefore recommended for consistently generating a product of optimum quality. Operating laboratory steam refining/deodorization outside of the optimum region will result in oil of variable quality. This is particularly important when standard laboratory studies are conducted to assess the downstream effects of other processing operations (*e.g.*, crude oil extraction, refining and bleaching techniques) on finished oil quality.

Pilot-plant batch deodorization. The purpose of the pilot-plant experiments was to determine the steam refining/deodorization parameters for the feed oil produced through either alkali or physical refining. To define acceptable batch processing parameters for a broad range of feed oils, the variable parameters included the level of FFA in feedstock, steam rate and temperature. Pilot-plant batch steam stripping was investigated with a 2^3 factorial experimental design with a replicated centerpoint (Fig. 1), which is capable of generating a mathematical model that contains not only the linear effect of the variables (X_1, X_2, X_3), but also their interactions (*e.g.*, X_1X_2), as follows:

$$Y = A_0 + A_1X_1 + A_2X_2 + A_3X_3 + A_4X_1X_2 + A_5X_1X_3 + A_6X_2X_3 \quad [6]$$

Because the pilot-plant experiment encompassed three variable parameters (FFA level in feed oil, steam rate and temperature), the design required a total of 10 random-order tests, including all possible high/low combinations of parameters and two replications of centerpoint conditions.

The actual ranges of the variable parameters were: temperature 420–500°F (219–260°C), sparging steam rate 1.0–4.0%/h and feed oil FFA 0.1–3.2% (Fig. 1). The remaining factors (feed oil type, batch size, pressure, heating/cooling gradient, processing aids, time) were constant.

To obtain feedstocks representing a range from low to high acidity, alkali-refined, bleached, dewaxed corn oil was blended with the required quantity of commercial corn oil fatty acids (Holm, Welch & Clark, Newark, NJ) to produce oils containing 0.1%, 1.65% and 3.2% FFA, respectively.

Table 2 shows the characteristics of finished, steam-refined/deodorized corn oils. The highest temperature tested [500°F (260°C)] produced lighter oil as a result of a greater degree of "heat bleaching". After 90 min, deacidification of all oils attained a satisfactory level (<0.040% FFA) except for the oils produced from high FFA feedstock processed at low temperature [3.2% FFA & 420°F (216°C), shown in bold]. Tocopherol retention proved sensitive to the process parameters employed. After only 90 min, high temperature/high sparging steam rates led to unacceptable depletion of tocopherols to levels below 0.08% (absolute content, shown in bold), which is recognized as a minimum for oil stability. This demonstrates that a careful selection of steam refining/deodorization parameters for a specific feedstock is necessary to produce a high-quality finished oil. Statistical interpretation of experimental data established time-related models for red color (COL), residual FFA and tocopherol retention (TOC):

TABLE 2

Pilot-Plant[a] Batch Steam Refined/Deodorized Corn Oil

Parameters (f/t/s)	Color (Y/R)	FFA (%) at 90 min	FFA (%) at 120 min	FFA (%) at 150 min	Tocopherols (%) at 90 min	Tocopherols (%) at 120 min	Tocopherols (%) at 150 min
0.1/420/1	8.2/1.6	0.030	0.030	0.020	0.130	0.129	0.123
0.1/420/4	7.9/1.5	0.020	0.020	0.010	0.119	0.117	0.103
0.1/500/1	6.5/1.3	0.030	0.030	0.020	**0.085**	**0.072**	**0.069**
0.1/500/4	5.7/1.2	0.040	0.040	0.030	**0.054**	**0.042**	**0.034**
1.6/460/2.5	7.9/1.6	0.040	0.030	0.030	0.106	0.101	0.089
1.6/460/2.5	7.7/1.5	0.040	0.020	0.020	0.108	0.102	0.090
3.2/420/1	12.1/2.0	**0.400**	**0.200**	**0.120**	0.111	0.108	0.101
3.2/420/4	8.6/1.7	**0.070**	0.030	0.020	0.116	0.103	0.099
3.2/500/1	5.8/1.4	0.030	0.030	0.020	**0.067**	**0.057**	**0.052**
3.2/500/4	7.6/1.2	0.030	0.030	0.030	**0.032**	**0.027**	**0.022**

[a]Constant parameters: 90 lb oil (3.8R in 5.25-inch cell, 0.130% tocopherol), 1.8–2.0 mmHg absolute pressure, 2 h.

	R^2	Adj. R^2	
$COL_{150},R = 3.085 + 0.586f - 0.003t - 0.026fs - 0.001ft$	0.959	0.926	[7]
$FFA_{90},\% = 0.018 + 0.938f - 0.215fs - 0.002ft + 0.0004fts$	0.975	0.955	[8]
$FFA_{120},\% = 0.023 + 0.443f - 0.111fs - 0.0009ft + 0.0002fts$	0.948	0.907	[9]
$FFA_{150},\% = 0.0173 + 0.266f - 0.071fs - 0.0005ft + 0.0001fts$	0.958	0.925	[10]
$TOC_{90},\% = 241.870 - 3.846f - 0.332t + 39.613s - 0.096ts$	0.933	0.880	[11]
$TOC_{120},\% = 371.405 - 4.031f - 0.622t - 4.936s$	0.917	0.876	[12]
$TOC_{150},\% = 341.158 - 3.413f - 0.567t - 0.012ts$	0.941	0.912	[13]

where: R = red color; s = sparging steam rate, %/h; f = feed oil FFA, %; subscripts = sampling time, minutes.

The response surfaces generated by equations 7-13 are presented in Figure 4. They indicate that for 0.1, 1.65 and 3.2% FFA feedstocks and 150-min steam-refining/deodorization time, an optimum temperature lies between 440-460°F (227-238°C) and a sparging steam rate between 3.0-2.5%/h.

Oil stability, evaluated by color reversion and flavor development during dark-storage tests (Fig. 5), confirmed that the median steam/temperature processing parameters produced the most stable oil. Also, low temperature and high stripping steam rates tend to produce oil with good flavor stability. These conditions, however, would be undesirable on a commercial scale because of process

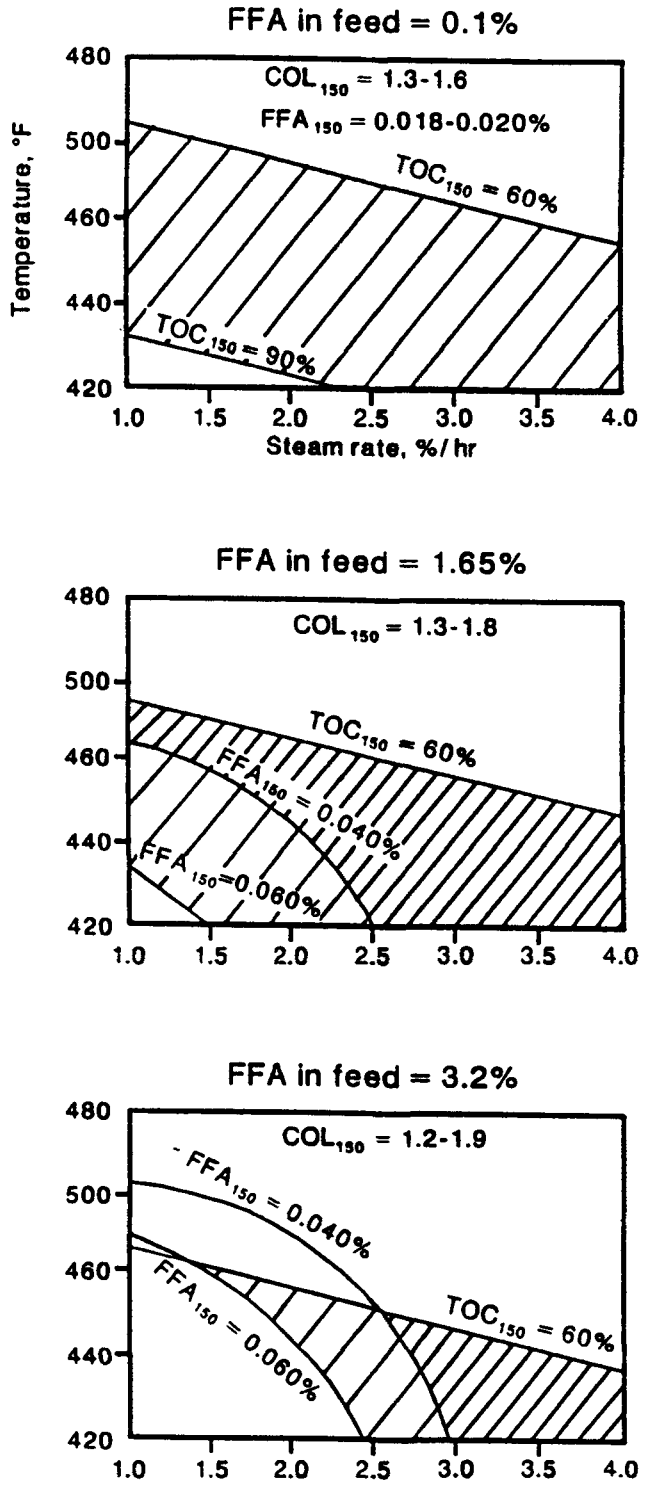

FIG. 4. Pilot-plant steam refining/deodorization—response surfaces.

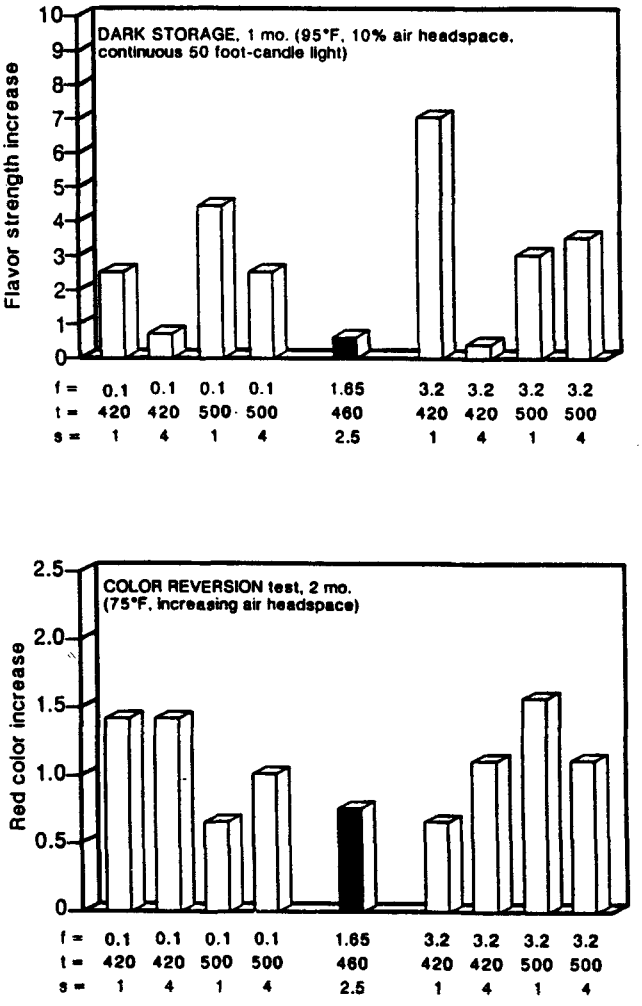

FIG. 5. Stability of pilot-plant steam refined/deodorized corn oil.

TABLE 3

Plant[a] Deodorized Soybean Oil

Variable parameters:						
Temperature, °F	475	475	488	488	500	500
Oil flow rate, lb/h	40,000	50,000	45,000	45,000	40,000	50,000
Oil characteristics:						
FFA (%)	0.020	0.024	0.018	0.018	0.014	0.017
Color (Y/R)	4.7/0.6	5.2/0.6	3.8/0.5	3.7/0.5	3.3/0.5	3.4/0.5
Peroxide value (meq/kg)	0	0	0	0	0	0
Anisidine value	1.7	2.2	1.9	2.4	1.9	2.0
Pesticides (ppm)	n.d.	n.d.	n.d.	n.d.	n.d.	n.d.
Tocopherols (%)	0.115	0.119	0.111	0.112	0.100	0.113
GC Volatiles (ppm)	2.5	1.9	2.6	1.8	1.5	1.7

[a]Constant parameters: semirefined oil (2.7R in 5.25-inch cell, 0.070% FFA, 0.136% tocopherol), absolute pressure 4.0–4.3 mmHg, sparging steam flow rate 880 lb/h. Abbreviations: FFA, free fatty acid; GC, gas chromatography; n.d., not detected.

economics: at higher sparging steam rates, the oil loss resulting from entrainment increases directly with the 6th power of the linear velocity of the vapor phase, which is directly related to the steam rate (1).

Because the level of retained tocopherol in the finished oil is the most reliable indicator of a properly optimized deodorization process, we tested the accuracy of the model equations on several oil types. Generally, the actual tocopherol retention varied less than 3% from the predicted values, validating our approach to process optimization.

Plant continuous deodorization. The objective of the plant experiment was to evaluate the effect of oil throughput rate and temperature on finished-oil quality. A 2^2 factorial design, based on the oil flow rate (40,000–50,000 lb/h) and the deodorization temperature [475–500°F (246–260°C)] as variable parameters, was used (Fig. 1) to investigate the effect on tocopherol retention and other measures of oil quality. Deodorizer feed-oil type, total stripping steam rate (880 lb/h) and column absolute pressure (4.0–4.3 mm Hg) were kept constant.

As each experimental point was run for 4 h, the total oil consumption for the design was over 1 million pounds. Because failure points were not desired for economic reasons, variable parameters' ranges were selected to produce only deodorized oil of acceptable quality.

Analytical results from the three samples taken per test point during steady-state operation were averaged, yielding the values given in Table 3. Besides routine quality-control indicators (*e.g.*, FFA, color, PV and AV), the oils were analyzed for tocopherols, pesticide residues and gas chromatography volatiles including pentane, pentanal, decadienal and other degradation products.

No pesticide residues were found in any of the experimental oils, which is consistent with our previous data (3,4). Except for pronounced differences in tocopherol level, all finished oils exhibited similar quality characteristics. The regression analysis for TOC produced the following equation:

	R^2	Adj. R^2	
$TOC = 821.255 - 12.840r - 1.569t + 0.028tr$	0.997	0.993	[14]

where: r = oil throughput rate in 1,000 lb/h; t = deodorization temperature, °F.

The response surface lots for tocopherol retention are shown in Figure 6.

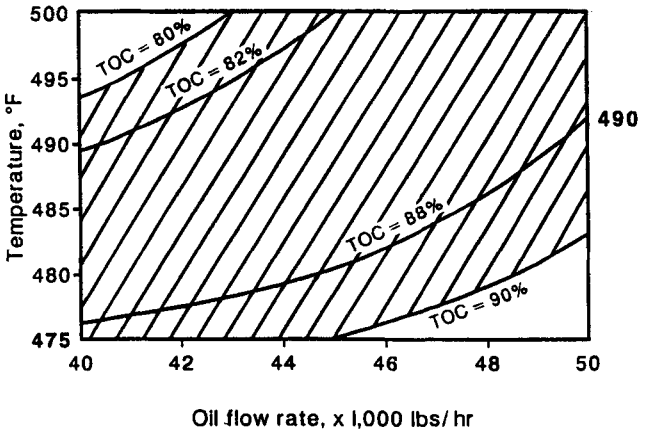

FIG. 6. Plant deodorization optimization—tocopherol retention.

While all experimental oils produced were of acceptable quality, those with 82 to 88% tocopherol retention were preferred. This suggests that optimum deodorization of soybean oil in the equipment evaluated requires 475 to 492°F (246–256°C) for the respective oil flow rates of 40,000 to 50,000 lb/h.

ACKNOWLEDGMENTS

The authors gratefully acknowledge the contribution of experimental data by G.F. Winnie, analytical data by M.A. Woodruff and V.L. Batandolo, and the statistical evaluation by C.E. Fritz.

REFERENCES

1. Norris, F.A., in *Bailey's Industrial Oil and Fat Products*, edited by T.H. Applewhite, Vol. 3, John Wiley & Sons, New York, 1979, pp. 127–167.
2. *Official and Tentative Methods of the American Oil Chemists' Society*, 3rd edn., edited by D. Firestone, Champaign, 1991.
3. Strecker, L.R., J.M. Hasman and A. Maza, in *Proceedings of the World Conference on Emerging Technologies in the Fats and Oils Industry*, edited by A.R. Baldwin, American Oil Chemists' Society, Champaign, 1986, pp. 51–55.
4. Strecker, L.R., A. Maza and G.F. Winnie in *Proceedings of the World Conference on Edible Fats and Oils: Basic Principles and Modern Practices*, edited by D.R. Erickson, American Oil Chemists' Society, Champaign, 1989, pp. 309–323.
5. Stolp, K.D., and R.W. Stute, U.S. Patent 4,341,713 (1982).

[Received October 21, 1991; accepted July 18, 1992]