Application of Two Heating Methods in Physical Refining of High-FA Olive and Sunflower Oils

J.C. Bada Gancedo[a,*], **Mª M. Prieto González**[c], **J. Xiberta Bernat**[d], **E. Graciani Constante**[b], **and M. León Camacho**[b]

[a]Instituto de Productos Lácteos de Asturias, Consejo Superior de Investigaciones Científicas, Villaviciosa, Asturias, Spain,
[b]Instituto de la Grasa y sus Derivados, Consejo Superior de Investigaciones Científicas, E-41012 Sevilla, Spain,
[c]Higher School of Industrial Engineering, E-33204 Gijón, Asturias, Spain, and
[d]Escuela Téncica Superior de Ingenieros de Minas de Oviedo, E-33004 Oviedo, Asturias, Spain

ABSTRACT: Two classes of vegetable oils, olive and sunflower, were processed by physical refining in a pilot plant with a capacity of up to 30 L by means of discontinuous deodorization, and distillates were recovered by condensing and freezing using steam and nitrogen as stripping gases. Two heating systems were evaluated in the deodorizer. In the first, the deodorizer oil and the distilled gases were heated so as to maintain the same temperature in both. In the second, only the oil was heated, resulting in a difference in temperature between the oil and the distilled gases. In addition, two different oil temperatures were evaluated for each stripping gas. By means of the first heating system, the deacidification time for both oils was reduced and the efficiency of the process was notably improved. On the other hand, the higher temperature had a negative influence over both parameters. For both heating systems the sterol contents did not suffer significant variations. Substantial variations in *trans* FA were not observed, and the composition of FA remained stable except for linoleic acid, which decreased, although more slowly than when the temperature was not maintained, as a result of the rapid formation of its *trans* FA.

Paper no. J9969 in *JAOCS 79*, 209–214 (March 2002).

KEY WORDS: Deodorization, deodorizer distillate, distillates, free fatty acids, olive oil, physical refining, sterols, sunflower oil, *trans* fatty acids.

The deodorization of edible vegetable oils in a batch process is a function of the following parameters: the working temperature of the oil, the absolute pressure at the head of the deodorizer (gas zone), the stripping gas flow rate, and the height of the oil layer, which conditions the trajectory of the stripping gas bubbles within the deodorizer. The free space between the surface of the oil and the gas exit allows some of the stripped droplets to fall back into the oil due to gravity. At the same time, distilled droplets condense on the colder surfaces within the trajectory to the exit. Traditionally, dry saturated steam is used as stripping gas, and although the use of nitrogen was proposed a number of years ago (1), it has only recently been tested as a stripping gas (with highly successful results) in a laboratory-scale deodorizer (2,3), at pilot-plant scale (4,5), and even at industrial scale (6).

The influence of the exit temperature of the deodorizer gases on the efficiency of deodorization was demonstrated by Deffense (7) after carrying out laboratory evaluations with soybean oil enriched with stearic acid. Prieto González *et al.* (8) evaluated two heating systems in a pilot-plant deodorizer to study the effect of exit temperature of the gases. Nitrogen was used as stripping gas, and two types of mixtures of oils were employed: (i) refined commercial sunflower oil with 99.9% pure oleic acid and (ii) the same sunflower oil with liquid distillates from the deodorization of olive oil. Maintaining the temperature of the gases exiting the deodorizer increased the deodorization rate and increased process efficiency.

On the basis of the previous satisfactory results, the present research was undertaken. The same method was applied to two vegetable oils—virgin olive oil and sunflower oil—in order to corroborate the results of the prior study and to approach as near as possible the conditions of industrial plants. Two types of stripping gas were employed: dry water vapor and food-grade nitrogen.

They determined the deacidification rate (FFADR), the final acidity of the distillates collected in the FA condenser (FFAC), the efficiency of the process (E), and the composition of the sterol fraction.

EXPERIMENTAL PROCEDURES

Description of the pilot plant. Physical refining was carried out in the pilot plant at the Higher School of Industrial Engineering (ETSII) in Gijón (University of Oviedo, Spain), using a deodorizer constructed of AISI 316-L stainless steel (8,9).

The installation consisted of a 30-L capacity cylindrical deodorizer (250 mm × 600 mm), the base being a semispherical cap with a flat top and a 120-mm torus ring at the bottom with 0.5-mm diameter holes drilled at a 45º angle downward and through which either nitrogen or steam from a steam generator, with a heat-generating system of a set of electrical elements, entered. The oil was electrically heated by means of

---

*To whom correspondence should be addressed at Instituto de Productos Lácteos de Asturia, C.S.I.C., Aptdo 85, E-33300 Villaviciosa, Asturias, Spain. E-mail: jcb@ipla.csic.es ceramic clamps installed on the external casing of the deodorizer. The distilled gases were heated by means of heating strips situated in the space for gases in the deodorizer and in the exit tube for the gases. Below the deodorizer were a cylindrical cooling unit for the finished oil, with a water-cooled coil, and a water-cooled shell and tube FA condenser in which most of the gaseous distillates were recovered, and a vertical shell and tube freezer refrigerated with silicon oil at –40°C, in which the remaining gases, mainly consisting of sterols, tocopherols, and hydrocarbons that were not previously condensed, were frozen. A cylindrical drop separator with two vertical stages was present, in which any possible unfrozen remains were condensed, thus avoiding their entry into the vacuum pump, as well as a vacuum pump (model PIL-11; MPR, San Sebastián, Spain) capable of generating the necessary vacuum with a capacity of 100 m^3/h. The entire installation, except for the cooler, was thermally insulated with 60-mm-thick glass wool and equipped with an automatic apparatus for registering the temperature and the working pressure.

Description of the deodorization trials. Sixteen trials, divided into two sets of eight trials each, were carried out, using 13 L oil in each assay. In the first set of trials, raw olive oil with a free acidity of 7.4% (measured as wt% oleic acid) supplied by the cooperative Jaencooperativa (Villanueva del Arzobispo, Jaén, Spain) was used. This set of trials was divided into two subsets, using nitrogen as stripping gas in four assays and steam in the other four. In two trials of each subset (one with heating of the exit gases and the other without), the oil temperature was 245°C, and in the other two, 265°C. A mass flow rate of 22.5 g/h was employed in the trials with nitrogen. In the trials with steam, 65 g/h of water vapor was employed for olive oil and 56 g/h for sunflower oil.

The second set of eight trials was carried out with raw sunflower oil, supplied by SIMSA (Pontejos, Santander, Spain), repeating the same protocol with the same parameters as those used for the olive oil of the first set.

The procedure of physical refining employed was the following: Once the required pressure (between 3 and 5 mm Hg) and the temperature inside the deodorizer reached 90ºC, the deodorizer was loaded with oil by vacuum aspirating from a tank. The oil was then heated, and a small amount of stripping gas was injected (8 L/h) beginning at 100°C in order to stir the oil and to avoid localized overheating that would cause oil damage. Upon reaching 180°C, the full mass flow of gas was injected. At 225°C, a sample was extracted and its acidity was determined; this point was designated as $t < 0$. On reaching the working temperature, a sample was extracted every hour, this instant being designated as $t = 0$. Once deodorization was completed, a sample was collected of the distillates condensed in the FA condenser and in the freezer. The entire installation was emptied and cleaned with steam and then dried with compressed air.

Methods. The rate of deacidification was determined in all the trials by measuring the acidity of the oil during the process of deodorization, and the final acidity of the distillated gases (in the condensed and in the frozen distillates). Obtained were the final acidity, the percentage composition of FA in the oil, the percentage composition of *trans* FA, and the sterol content. For the acidity analysis, the AOCS standard method (10) was followed. The influence of the deodorization temperature and of the heating systems in the formation of *trans* FA was also studied.

The efficiency of the process was likewise calculated in each trial. In order to study possible variations in the percentage composition of the FA, which is traditionally assumed to be similar to that of the initial oil, this variable was analyzed in each sample of each deodorization trial.

The acidity of the oil in the deodorizer as a function of time was approximated by the exponential mathematical expression $y(t) = A \cdot e^{-kt}$, in which $y(t)$ represents the acidity of the oil at time t and k corresponds to FFADR. This value gives a measurement of the effectiveness of the deodorization: The lower the FFADR, the greater the time needed to attain a specific final acidity, and vice versa. The deodorization efficiency, measured as the vaporization efficiency, was expressed by Vian's formula (11) and by that described by Andersen (12). The acidity of the distillates collected in the FA condenser supplies the effectiveness of recovery of the oil stripped by the gases on exiting and is thus an indicator of the losses produced in the process. The greater the acidity, the lower the amount of stripped neutral oil.

The statistical technique of blocking with paired comparisons (13) was employed to analyze the results and to establish the influence of the heating system, the temperature of the oil in the deodorizer, and the amount of stripping gas on the variables selected to describe the deodorization process.

RESULTS AND DISCUSSION

In the identification of each trial, the asterisk (*) indicates that heating was carried out (UH = 1). When it is not present, this means that there was no heating (UH = 0). Table 1 presents the working conditions for each trial, as well as the different values for acidity of the olive and sunflower oils during the period of deodorization for UH = 1 and UH = 0.

For UH = 1, the average pressure inside the deodorizer varied ± 1 mm Hg, and the temperature of the oil varied ±1.5°C with respect to the preestablished temperature. Likewise, the exit temperature of the distilled gases varied ±2°C with respect to the preestablished temperature of each trial.

For UH = 0, the average pressure varied ± 1 mm Hg, and the temperature of the oil varied ±1.5°C with respect to the preestablished working temperature. The exit temperatures of the gases were: for the olive oil, 173 ± 3°C for the trials at 245°C, and 205 ± 3°C for the assays at 265°C; for the sunflower oil, 150 ± 3°C for the trials at 245°C, and 175 ± 3°C for the trials at 265°C.

A slight decrease was observed in the acidity of the olive oil from $t < 0$ until $t = 0$, due to the fact that deodorization commenced before the preestablished temperature was reached. A comparison of the values shows that: (i) when steam was employed, deacidification was always faster than

TABLE 1
Processing Conditions and Acidity in the Deodorizer (olive and sunflower oils)

					Acidity (%)									
Trial	Stripping gas	m_{gas} (g/h)	T (°C)	P (mm Hg)	$t_{<0h}$	0 h	1 h	2 h	3 h	4 h	5 h	6 h	7 h	8 h
Olive oil														
E41	Steam	65	245	3.2	6.72	6.48	5.21	4.36	2.00	1.00	0.43	0.20	—	—
E39[a]	Steam			4.5	4.11	2.30	0.87	0.20	0.15	—	—	—	—	—
E43	Steam		265	4.2	6.83	3.32	1.60	0.98	0.38	0.19	0.15	—	—	—
E40[a]	Steam			4.9	6.87	4.44	0.66	0.20	0.14	—	—	—	—	—
E37	N_2	22.5	245	6.5	7.60	6.97	6.68	6.29	5.74	5.39	5.18	4.95	—	—
E38[a]	N_2			7.3	6.99	6.68	6.45	5.92	5.11	4.22	3.77	3.50	—	—
E36	N_2		265	7.0	6.80	5.39	4.07	3.74	3.57	3.43	3.34	3.12	2.95	—
E35[a]	N_2			6.2	7.08	4.76	2.30	1.85	1.74	1.72	1.72	1.66	1.67	—
Sunflower oil														
E45	Steam	56	245	4.8	1.42	1.37	0.94	0.59	0.40	0.37	0.29	0.24	0.17	—
E49[a]	Steam			5.8	1.44	1.30	1.11	0.70	0.38	0.18	—	—	—	—
E47	Steam		265	4.4	1.35	1.30	0.97	0.72	0.58	0.53	0.58	0.60	0.65	—
E48[a]	Steam			5.1	1.45	1.21	1.00	0.52	0.25	0.19	—	—	—	—
E52	N_2	22.5	245	1.4	1.50	1.50	1.43	1.10	1.07	1.01	0.9	0.76	—	—
E55[a]	N_2			3.4	1.54	1.50	1.30	1.30	1.30	1.22	1.22	1.10	1.05	0.87
E53	N_2		265	4.1	1.63	1.61	1.39	1.29	1.26	1.22	1.16	1.15	1.10	—
E54[a]	N_2			3.5	1.60	1.48	1.34	1.32	1.04	0.76	0.68	0.56	0.45	0.42

[a] Trials carried out with heating of the exit gases (UH = 1); m_{gas}, mass flow rate of stripping gas; T, assay temperature; P, pressure at the head of the deodorizer; $t_{<0h}$, time to reach $T = 225°C$; 0 h, time to reach the assay T; heating rate 1.7°C/min.

with nitrogen because the amount of steam was greater than that of nitrogen and (ii) when the exiting distilled gases were heated, the speed of deacidification was greater than when there was no heating, when maintaining the same amount of stripping gas, due to the decrease in refluxing distilled droplets to the deodorizer because their condensation in the exit stage was avoided.

Table 2 presents a comparison of FFADR, FFAC, and E results with UH = 0 and UH = 1 for each trial and type of oil. The values of FFADR were substantially higher for UH = 1 than for UH = 0, and the efficiency quotients were considerably greater than unity. Those values indicate the positive effects on the process of heating the distilled gases. On the other hand, the values of FFAC demonstrate that stripping of neutral oil occurred, being substantially lower when nitrogen was injected than when injecting steam, which diminishes the losses in deodorization and increases the purity of the condensed distillates. Heating the exit gases had a slightly beneficial effect on FFAC values.

Tables 3 and 4 present the mean and SD of the percentages of each FA in the samples of olive and sunflower oils, respectively. The percentage of each FA was quite stable, except for linoleic acid, which decreased moderately. This change may be due to the rapid formation of its *trans* FA.

Table 5 presents the variations throughout the process of the contents of *trans* FA and sterols for olive oil. No significant modifications were observed for sunflower oil. The formation of *trans* FA was lower for UH = 1 and for steam. The

TABLE 2
Comparison of the Results with UH = 0 and UH = 1 for Raw Olive and Sunflower Oils[a]

Trial		T	m_{gas}			$FFAC_{UH=1}$	$FFAC_{UH=0}$	
UH = 1	UH = 0	(°C)	(g/h)	$FFADR_{UH=1}$	$FFADR_{UH=0}$	(%)	(%)	$E_{UH=1}/E_{UH=0}$
Olive								
E39	E41	245	65	0.879	0.684	58	36	1.989
E38	E37		22.5	0.131	0.061	72	48	1.862
E40	E43	265	65	0.775	0.637	54	50	1.902
E35	E36		22.5	0.053	0.049	90	56	1.365
Sunflower								
E49	E45	245	56	0.607	0.258	62	16	2.052
E55	E52		22.5	0.031	0.109	79	84	1.503
E48	E47	265	56	0.571	0.090	55	34	1.889
E54	E53		22.5	0.190	0.037	83	80	2.072

[a] The values of the deodorization variables (FFADR and FFAC) and the quotients of E are compared for equal oil temperatures and stripping gas mass flow rates with respect to the heating method (UH = 1 and = 0); T, processing temperature; m_{gas}, nitrogen mass flow rate; FFADR, FA distillation rate, calculated from $t = 1$ h after reaching the processing temperature; FFAC, free FA content; E, free FA vaporization efficiency.

TABLE 3
Statistical Results of Percentages of Each FA of the Trials (olive oil)

		Palmitic (%)		Stearic (%)		Oleic (%)		Linoleic (%)	
Trial		$\bar{y}$	*s*	$\bar{y}$	*s*	$\bar{y}$	*s*	$\bar{y}$	*s*
E41	Deod.	10.16	0.11	2.98	0.08	77.73	0.95	3.86	0.21
	Cond.	11.15	0.52	3.10	0.04	74.24	—	3.58	0.11
E39[a]	Deod.	10.13	0.22	3.00	0.08	77.11	0.58	3.94	0.25
	Cond.	10.65	—	1.66	—	75.91	—	4.21	—
E43	Deod.	10.02	0.23	3.09	0.10	77.49	0.45	3.57	0.47
	Cond.	10.88	—	2.90	0.03	75.92	—	3.78	0.22
E40[a]	Deod.	10.15	0.23	3.04	0.08	77.10	0.64	3.48	0.71
	Cond.	10.11	0.17	2.86	0.11	76.55	—	3.60	0.33
E37	Deod.	10.30	0.35	3.23	0.14	76.47	0.57	3.12	0.54
	Cond.	—	—	—	—	—	—	—	—
E38[a]	Deod.	10.67	0.37	2.96	0.12	77.21	0.64	3.38	0.59
	Cond.	10.17	—	3.04	—	76.07	—	3.98	—
E36	Deod.	10.80	0.63	3.10	0.21	76.19	1.42	2.22	1.08
	Cond.	10.75	—	2.94	—	74.59	—	3.62	—
E35[a]	Deod.	10.47	0.26	3.13	0.10	76.95	0.55	2.34	0.63
	Cond.	10.35	0.42	2.24	—	71.06	—	2.17	—

[a] Trials carried out with heating of the exit gases (UH = 1); $\bar{y}$, sample mean; *s*, SD of the sample; Deod., samples taken from the deodorizer; Cond., samples taken from the FA condenser.

TABLE 4
Statistical Results of Percentages of Each FA of the Trials (sunflower oil)

		Palmitic (%)		Stearic (%)		Oleic (%)		Linoleic (%)	
Trial		$\bar{y}$	*s*	$\bar{y}$	*s*	$\bar{y}$	*s*	$\bar{y}$	*s*
E49[a]	Deod.	7.27	0.17	5.70	0.21	25.83	0.68	52.54	2.98
	Cond.	11.57	—	6.24	—	30.22	—	39.94	—
E48[a]	Deod.	7.39	0.19	5.84	0.19	26.24	0.57	49.37	5.01
	Cond.	8.93	—	5.30	—	23.76	0.42	45.77	0.66
E52	Deod.	6.56	0.04	5.00	0.07	23.54	0.11	58.98	1.50
	Cond.	—	—	—	—	—	—	—	—
E55[a]	Deod.	6.90	0.19	5.32	0.24	24.66	0.66	54.66	3.74
	Cond.	12.54	—	5.37	0.04	28.15	—	45.60	2.57
E53	Deod.	6.77	0.06	5.14	0.08	23.77	1.01	51.01	8.08
	Cond.	—	—	—	—	—	—	—	—
E54[a]	Deod.	6.70	0.08	5.25	0.11	24.26	0.24	48.74	9.68
	Cond.	9.76	—	3.45	—	22.80	—	45.17	4.51

[a] Trials carried out with heating of the exit gases (UH = 1); $\bar{y}$, sample mean; *s*, SD of the sample; Deod., samples taken from the deodorizer; Cond., samples taken from the FA condenser.

TABLE 5
Content in *trans* FA of the Olive Oil (% of the total FFA)

	Stripping	m_{gas}	T	P	*Trans* fatty acids (%)			
Trial	gas	(g/h)	(°C)	(mm Hg)		O^t	$L^{c,t}$	$L^{t,c}$
E41	Steam	65	245	3.2	Initial	0.30	0	0
					Final	0.67	0.12	0.12
E39[a]	Steam	65	245	4.5	Initial	0.31	0	0
					Final	0.59	0.12	0.12
E43	Steam	65	265	4.2	Initial	0.32	0	0
					Final	0.95	0.33	0.33
E40	Steam	65	265	4.9	Initial	0.32	0	0
					Final	0.95	0.33	0.33
E37	N_2	22.5	245	6.5	Initial	0.32	0	0
					Final	0.95	0.33	0.33
E38	N_2	22.5	245	7.3	Initial	0.32	0	0
					Final	0.95	0.33	0.33
E36	N_2	22.5	265	7.0	Initial	0.32	0	0
					Final	0.95	0.33	0.33
E35	N_2	22.5	265	6.2	Initial	0.32	0	0
					Final	0.95	0.33	0.33

[a] Trials carried out with heating of the exit gases (UH=1); m_{gas}, stripping gas mass flow rate; T, processing temperature; P, pressure at the head of the deodorizer; O^t, isomer 9*t* of oleic acid; $L^{c,t}$, isomer 9*t*,12*c* of linoleic acid; $L^{t,c}$, isomer 9*c*,12*t*; initial, initial content; final, content at the end of the process.

TABLE 6
Sterols Content at the Beginning and End of the Deodorization Process in Olive Oil (ppm)

Trial		Sterols (ppm)				
		Camp.	β-Sit.	Δ5-Av.	Others	Total
E41	Initial	74	1570 ± 170	96	83	1823 ± 155
	Final	69	1540 ± 166	96	106	1811 ± 154
E39[a]	Initial	59	1251 ± 135	65	78	1453 ± 124
	Final	61	1349 ± 146	76	89	1575 ± 134
E43	Initial	76	1544 ± 167	98	129	1847 ± 157
	Final	49	1090 ± 118	65	80	1284 ± 109
E40[a]	Initial	70	1662 ± 179	99	109	1940 ± 165
	Final	52	1152 ± 124	70	84	1358 ± 115
E37	Initial	79	1272 ± 137	56	94	1501 ± 128
	Final	60	1314 ± 142	67	65	1506 ± 128
E38[a]	Initial	56	1065 ± 115	62	67	1250 ± 106
	Final	58	1275 ± 138	71	64	1468 ± 125
E36	Initial	49	989 ± 108	51	82	1171 ± 100
	Final	55	1086 ± 118	62	112	1315 ± 112
E35[a]	Initial	57	1242 ± 134	64	62	1425 ± 121
	Final	60	991 ± 107	57	49	1157 ± 98

[a] Trials carried out with heating of the exit gases (UH = 1); camp., campesterol; β-sit., β-sitosterol; Δ5-av., Δ5-avenasterol; others: cholesterol, campestanol, stigmasterol, Δ7-campesterol, chlerosterol, sitostanol, Δ-5,24 stigmastadiol, Δ7-stigmasterol; total: total sterols content; initial: initial content; final: final content.

absolute values of the *trans* FA in olive oil were higher than those contained in the European Union norm (14), because we started out with an oil having a high initial *trans* FA content and high acidity.

Table 6 presents the values of the most interesting sterols for olive oil, as well as their total amounts. The AOCS standards and the statistical method (15) were applied to calculate methodological errors.

Table 7, in which m_{gas} represents the mass flow rate of steam consumed and the mass flow rate of nitrogen expressed in relation to equivalent steam in accordance with Graciani Constante *et al.* (16), presents the values of the coefficients of significance for the influence of the process variables on the parameters of the process, applying the statistical technique of blocking with paired comparisons. The process efficiency was adversely affected by increased mass flow rate of the stripping gas and by heating the deodorization gases. Temperature and heating increased the acidity of the condensates collected during the process, and an increase in the stripping gas flow rate reduced the acidities of the condensates. The total sterols content decreased slightly only when increasing the temperature or stripping gas flow rate and was not affected by heating system. The speed of deacidification was favored by heating the exit gases, and by the increase of the stripping gas flow rate, decreasing with the temperature of the oil. For sunflower oil, differences in that temperature of the oil had no significant effects.

TABLE 7
Significance Coefficients of the Influence of the Process Parameters on the Process Variables in Olive and Sunflower Oils (blocked pairs method)

	UH	T	m_{gas}
	$(1-\alpha_c)\cdot 100$		
Olive			
FFADR	95.45	97.20	99.98
E	96.92	94.95	99.08
FFAC	97.82	92.14	95.96
SC	—	92.70	90.94
Sunflower			
FFADR	92.00	—	95.48
E	93.34	—	97.85
FFAC	—	—	98.11

[a] $(1-\alpha_c)\cdot 100$, significance coefficient; UH, heating; *T*, temperature; m_{gas}, stripping gas mass flow rate; FFADR, deacidification rate; E, efficiency; FFAC, acidity of the condensates; SC, sterols content.

ACKNOWLEDGMENTS

This study was financed by the CICYT (Proyecto ALI95-0517). We wish to thank Raúl Salcines for his technical assistance.

REFERENCES

1. Norris, F., Deodorization, in *Bailey's Industrial Oil and Fat Products*, edited by T.H. Applewhite, John Wiley & Sons, New York, 1985, Vol. 3, pp. 897–930.
2. Huesa Lope, J., and M.C. Dobarganes García, A Process for Deodorizing Fats and Oils, Patent No. ES 2,013,206 (1990).
3. Ruíz-Menéndez, M.V., G. Márquez-Ruíz, and M.C. Dobarganes, Comparative Performance of Steam and Nitrogen as Stripping Gas in Physical Refining of Edible Oils, *J. Am. Oil Chem. Soc. 73*:1641–1645 (1996).
4. Camacho, L., M.V. Ruíz-Méndez, and E. Graciani, Changes in Olive Oil Components During Deodorization and/or Physical Refining at Pilot Plant Scale Using Nitrogen as Stripping Gas, *Fett/Lipid 101*:38–43 (1999).
5. Graciani, E., J.C. Bada, F. Rodriguez, and M.V. Ruíz-Méndez, Physical Refining of Edible Oils Using Nitrogen as Stripping Gas. Process Optimization, *Grasas Aceites 45*:132–146 (1994).
6. Ruiz Méndez, M.V., Modificaciones producidas durante el proceso de refinación de los aceites comestibles, Ph.D. Thesis, University of Sevilla, Spain, 1993.

7. Deffense, E., Refinación con Vapor: Comparación de la Teoría con la Práctica, *Dossier Oleo*:49–57 (1994).
8. Prieto González, M.M., J.C. Bada, E. Graciani, and I. Lombardía, A Comparative Study of Two Heating Procedures in the Physical Refining of Edible Oils, *J. Am. Oil Chem. Soc. 76*:1471–1476 (1999).
9. Velázquez Vila, R., and J.C. Bada, *Producción de Alto Vacío Mediante Congelación de Condensables*, Comunicación a la XXII Asamblea de Miembros del Instituto de la Grasa y sus Derivados, CSIC, Sevilla, Spain, 1987.
10. *Official Methods and Recommended Practices of the American Oil Chemists' Society*, 5th edn., edited by D. Firestone, AOCS Press, Champaign, 1998, Method Cd 3d-63.
11. Vian, A., and J. Ocón, *Elementos de Ingeniería Química*, Aguilar, Madrid, Spain, 1964, p. 657.
12. Andersen, A.J.C., *Refinación de Aceites y Grasas*, edited by José Monteso, Barcelona, Spain, 1956, p. 280.
13. Box, G.E.P., W.G. Hunter, and J.S. Hunter, *Statistics for Experimenters. An Introduction to Design, Data Analysis and Model Building*, John Wiley & Sons, New York, 1978, pp. 93–106, 306–374.
14. EEC Comission Regulation No. 1429/92 of the 26th of May 1992 Which Modifies EEC Regulation No. 2568/91, *Off. J. Eur. Communities L 150*:17–20 (1991).
15. Vioque, A., and T. Albi, Residuos de pesticidas en grasas comestibles. I. Análisis de insecticidas clorados, *Grasas Aceites 23*:213–223 (1972).
16. Graciani Constante, E., F. Rodríguez Berbel, J. Huesa Lope, and A. Paredes Torronteras, Deacidification by Distillation Using Nitrogen as Stripper. Possible Application to the Refining of Edible Fats, *Ibid. 42*:286–292 (1991).

[Received May 4, 2001; accepted December 1, 2001]