A Comparative Study of Two Heating Procedures in the Physical Refining of Edible Oils

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ABSTRACT: Two mixtures of refined sunflower seed oil, one with oleic acid and the other with olive oil distillates from a laboratory plant, were physically refined using nitrogen as stripping gas in a discontinuous deodorization pilot-plant scale installation (30-L capacity). Two heating procedures were tested: one using independent electrical heating for the oil and the gas distillates so as to maintain the same temperature in both, and another in which only the oil was heated and controlled, resulting in a difference in temperatures in the oil and the gas distillates. Two different oil temperature values and three nitrogen flow rates were also assayed. The statistical technique of blocking with paired comparisons was used to analyze the results. These results showed that maintaining the same temperature in the oil and gas distillates had a positive effect on free fatty acid distillation rate and vaporization efficiency. Oil temperature and nitrogen flow rate also influenced some of the aforementioned responses.

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Operating conditions in vertical discontinuous deodorizers used in the refining of edible oils are described by means of the values inside the deodorizer: pressure in the zone of the phase-gas distillates, oil temperature, stripping gas flow, and height of the oil layer. The stripping gas that has traditionally been used is live steam, although the use of nitrogen as stripping gas has been suggested in the past (1), and the performance of both gases has been compared (2–4). The values of oil temperature, nitrogen flow rate, and height of the oil layer that lead to the best experimental results have been discussed in the physical refining of sunflower seed oil in a discontinuous deodorizer of 200-kg capacity (5). In this last paper, the following values were proposed: oil temperature from 250 to 255°C and nitrogen flow rate from 1.4 to 2.3 3/cubic meters per metric ton per hour, expressed at 1 bar pressure and 0°C.

With regard to the influence of the temperature distribution inside the deodorizer, some results were presented for a mixture of soya oil and stearic acid (4% w/w), using live steam as stripping gas (6). In this case, the assays were carried out in a glass discontinuous laboratory deodorizer of 2-L capacity. The deodorizer oil was placed inside an electrically heated furnace so as to obtain the same temperature in both the gas phase and the oil.

In this paper, the results of the physical refining of two mixtures are presented: (i) commercial, refined sunflower seed oil [initially 0.1% w/w of free fatty acids, (FFA)] and oleic acid; and (ii) the same sunflower seed oil and olive oil distillates from a laboratory refining plant. The assays were carried out in a pilot plant-scale installation made of stainless steel, which was specifically designed for this purpose. Two heating procedures were compared. In one, the same temperature was maintained in the gas distillates and the liquid (oil) using an electrical heating system that independently heated and controlled the temperature for each part of the deodorizer (i.e., gas and liquid zones); in the other, only the part occupied by the oil was heated and the oil temperature controlled. In both procedures, the temperatures of the oil and the gas distillates were continuously measured and registered. In addition, two values of oil temperature and three flow rates of nitrogen, used as stripping gas, were assayed. These values were chosen taking into account those suggested by Graciani *et al.* (5) and the limitations of the installation.

To describe the deodorization performance, the FFA distillation rate (FFADR), the vaporization efficiency of FFA (E), and the FFA content in the distillates (FFAC), once they had been recovered by condensation in a surface heat exchanger, were used.

The statistical technique of blocking with paired comparisons was used to analyze the results in order to establish the influence of the heating procedure, the oil temperature, and the flow rate of the stripping gas on the responses chosen to describe the deodorization performance.

FFADR. The data of FFA content in the deodorizer as a function of time can be approached by the exponential mathematical expression $y = Ae^{-Kt}$, where y is the FFA content in the oil at time t; A is the initial FFA content in the oil; and K is the FFADR. The FFADR gives a sort of measure of deodorization effectiveness; the lower the FFADR, the greater the time required to obtain a certain final FFA content in the deodorized oil, and vice versa.

Vaporization efficiency. This value is determined by the formula that was theoretically established for technological distillation operations with a stripping gas, in the case of high FFA contents (7),

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$$E_{1-2} = \frac{\dfrac{PO}{P_v}\ln\dfrac{\mathrm{FFA}_1}{\mathrm{FFA}_2} + \dfrac{P}{P_v}(\mathrm{FFA}_1 - \mathrm{FFA}_2)}{S + \mathrm{FFA}_1 - \mathrm{FFA}_2} \qquad [1]$$

where E_{1-2} is the vaporization efficiency of the FFA between time 1 and 2; P (mm Hg) is the pressure at the head of the deodorizer; P_v (mm Hg) is the vapor pressure of the major compound in the distillates; O is the number of moles of oil (880 g as average molecular weight); S is the number of moles of stripping gas; and FFA_1 and FFA_2 are the number of moles of the FFA at times 1 and 2, respectively (282 g as average molecular weight).

FFAC. FFAC indicates the effectiveness of drop recovery and the quantity of FFA recovered. The lower the FFAC, the greater the oil losses.

EXPERIMENTAL PROCEDURES

Description of the installation. Deodorization took place in a 30-L oil deodorizer made of AISI 316-L stainless steel. The deodorizer was a cylindrical vessel (250 × 600 mm) with a hemispherical bottom and flat top. An inlet tube with a stopcock led the incoming nitrogen to the bottom of the deodorizer, and the stripping gas was distributed through a perforated stainless-steel ring (120 mm diameter, 0.5 mm hole size). At the top of the deodorizer, before the gas distillate outlet, there was a drift eliminator that recovered the oil droplets. The oil to be deodorized was heated electrically by a ceramic bracket installed on the external surface of the vessel, and the gas distillates were heated by a heating strip.

The gas-phase distillates were partially condensed and recovered in a horizontal two-pass shell-and-tube heat exchanger refrigerated by water. The noncondensing gas was introduced into a vertical two-pass shell-and-tube heat exchanger refrigerated by a silicone oil with an inlet temperature of –40°C. The required vacuum was achieved by connecting the outlet of the vertical heat exchanger to a vacuum pump (model PIL6-11; MPR, San Sebastian, Spain). All the installation, except for the vacuum pump, was thermally isolated from the surrounding atmosphere by means of 60-mm-thick mineral fiberglass.

The temperature in the oil and the gas distillates and the pressure at the top of the deodorizer were measured and automatically registered. The temperatures were measured using PT-100 probes, calibrated in the range from 0 to 300°C with an accuracy of ± 1°C. The pressure was measured with a membrane measurement device (model 3051CA; Fisher-Rosemount, Chanhassen, MN), ±0.0075% of the span accuracy, calibrated between 0.5 and 20 mm Hg. The nitrogen flow rate was measured with a float area meter, ± 1 L/h sensitivity, calibrated from 0 to 100 L/h at 2 bar pressure and 20°C. The pressure of the injection of nitrogen was established using a pressure regulator calibrated between 1 and 7 bar (model 404; Union Carbide Gases, N.V., Olen, Belgium; ± 0.1 bar sensitivity). The temperature values in the oil and gas distillates were maintained, with an accuracy of ±1°C, at the established values using two controllers (model SR60; Shimaden Co., Ltd., Tokyo, Japan) that were connected separately to their corresponding PT-100 probe and electrical heating system.

Description of assays. In each assay, 13 L of commercial refined sunflower seed oil (Pulisol$^{\mathrm{TM}}$, Aceites Carrión, S.A., Toledo, Spain; initially 0.1% w/w of FFA) was employed. Two groups of assays were carried out. In the first, a mixture of sunflower seed oil and oleic acid (99.9% w/w purity) provided by Merck (Darmstadt, Germany) was refined. To check the results of this first set of assays when refining complex mixtures of distillates, a second set was performed. In this case, a mixture of the same sunflower seed oil and liquid distillates from olive oil deodorization (66% w/w of FFA), supplied by the Instituto de la Grasa de Sevilla (Seville, Spain), was refined. The initial FFA content in the mixtures was approximately 2.5% w/w.

Nitrogen was injected at 2 bar pressure and a temperature of 100°C, and the following flow rates were tested: 18, 30, and 47 L/h (values measured at 2 bar pressure and a temperature of 20°C). Assays were performed at 245 and 265°C, either maintaining the same temperature in the gas distillates and in the oil (heating the upper part of the deodorizer and controlling its temperature, UH = 1), or not maintaining the same temperature (no heating of the upper part of the deodorizer, UH = 0).

The oil was introduced into the deodorizer when the required vacuum was obtained in the installation (absolute pressure between 3 and 10 mm Hg). To minimize oil heating time and to avoid, as much as possible, vaporization outside of the operation conditions, the deodorizer vessel was heated until approximately 90°C before introducing the oil. At a temperature of about 100°C, a small nitrogen flow rate was injected to remove the oil, in order to prevent inappropriate wall temperatures. When the oil temperature reached 230°C, a first sample of oil was taken and FFA determined (the installation was equipped with a system for taking samples from the deodorizer and the condenser throughout refining without losing the appropriate vacuum). The time that corresponds with the measured FFA will subsequently be called *t* initial. When the required operation conditions were reached, at *t* = 0, the assay nitrogen flow rate was established, and a new sample of oil was taken to determine FFA. To ascertain the evolution of FFA with time, samples were taken hourly and their acidity determined according to AOCS standard methods (8) until complete, on 6 or 8 h under operating conditions.

RESULTS AND DISCUSSION

Tables 1 and 2 summarize the data of FFA evolution with time for UH = 1 and UH = 0, respectively. The first four columns indicate the additive added to make up the mixture, oil temperature, nitrogen flow rate, and assay denomination (a lowercase letter in the denomination indicates that this assay has been repeated), respectively. The data that correspond to the steadiest values in all the measured variables have been written in italics and have been used to calculate FFADR and E.

Case UH = 1. When adding oleic acid, the mean pressure in the deodorizer of the experiments was 4 mm Hg, a minimum value of 3.4 mm Hg being registered in E1a, and a maximum

TABLE 1
Free Fatty Acids Content vs. Time, with Heating of Gas Distillates (UH = 1)[a]

Additive	T(°C)	m_{N_2} (L/h)	Assay[b]	*t* initial	*t* = 0	*t* = 1	*t* = 2	*t* = 3	*t* = 4	*t* = 5	*t* = 6	*t* = 7	*t* = 8
Oleic acid	245	18	E1a	2.41	*1.91*	*1.65*	*1.64*	*1.42*	*1.03*	*0.76*	*0.55*	*0.41*	*0.26*
			E1b	2.47	2.30	*1.86*	*1.48*	*1.32*	*0.95*	*0.64*	*0.42*	*0.30*	0.25
		30	E2	2.30	2.26	*1.65*	*1.49*	*1.21*	*0.96*	*0.83*	*0.59*	*0.45*	*0.30*
		47	E3	2.33	*1.87*	*1.34*	*0.90*	*0.68*	*0.38*	*0.24*	*0.11*	*0.08*	—
	265	18	E4a	2.31	1.84	*1.22*	*0.73*	*0.32*	*0.21*	*0.21*	*0.21*	*0.18*	—
			E4b	2.33	2.00	*1.53*	*1.18*	*0.72*	*0.41*	*0.29*	*0.22*	*0.18*	—
		30	E5	2.47	2.16	*1.76*	*1.33*	*0.67*	*0.37*	*0.17*	*0.11*	*0.09*	—
		47	E6	2.43	1.77	1.14	*0.46*	*0.19*	*0.11*	*0.10*	*0.08*	*0.07*	—
Olive oil distillates	245	18	E7	2.38	2.14	*1.91*	*1.88*	*1.63*	*1.35*	*1.04*	*0.89*	—	—
	265	18	E8	2.42	2.4	*1.75*	*1.25*	*1.1*	*0.88*	*0.74*	*0.58*		

[a] The data that correspond to the steadiest values in all the measured variables have been written in italics and have been used to calculate free fatty acid distillation rate and vaporization efficiency of free fatty acids. m_{N_2}, nitrogen flow rate; *t*, time.
[b] Assay denomination. When a lowercase letter is appended, it indicates that this assay has been repeated.

value of 4.3 mm Hg in E6. The temperatures in the gas distillates were kept very close to the oil temperatures with differences of ±1°C. When olive oil distillates were added, the pressure in the deodorizer was close to 8 mm Hg. In E7, both temperatures, in the oil and in the gas distillates, were very close to the established values of 245°C, with differences of ±0.5°C. In addition, the temperatures in E8 were close to 265°C, with differences of ±1°C.

Case UH = 0. When adding oleic acid, the mean pressure in the deodorizer of the experiments was 4 mm Hg, a minimum value of 2.9 mm Hg being registered in E9 and a maximum value of 5.9 mm Hg in E14. In the assays, the temperatures in the oil were kept very close to the established values, with differences of ±0.5 and ±1°C for oil temperatures of 245 and 265°C, respectively. The measured temperatures in the gas distillates were around 66°C lower than the temperature to be maintained in the oil. The biggest temperature difference between the oil and the gas distillates (73°C) was registered in E10b, and the smallest (62°C) was registered in E11.

When olive oil distillates were added, the pressure in the deodorizer was close to 7 mm Hg in E15, and 8 mm Hg in E16. For E15, the temperature in the oil was 245 ± 0.5°C, and was 89°C lower in the gas distillates. For E16, the temperature in the oil was 265 ± 0.5°C and was 77°C lower in the gas distillates.

Comparing Tables 1 and 2, one can see that for all the assayed conditions, FFA variations are greater when the gas distillates are heated. This fact is related with the difference of temperature between the gas distillates and the oil. When UH = 0, the gas distillates temperature is lower than for oil. The FFA condense and can return to the oil (refluxing phenomenon) if the gas distillates temperature is under the temperature of saturation of the FFA. This refluxing could be avoided in part if the distillates were removed quickly, which could be done by increasing nitrogen flow rate. But keeping the same temperature in the oil and in the gas distillates would imply a good deodorization effectiveness with lower nitrogen flow rate (it is believed that the increase in the stripping gas flow rate facilitates distillate evacuation). Besides, the lower the stripping flow rate, the greater the efficiency (Eq. 1). Lower nitrogen flow rates would decrease oil loss by decreasing the drag force over oil drops, and the vacuum equipment would thus be smaller. These effects could represent important economic advantages.

By observing the oil temperature effects in each table separately, for equal nitrogen flow rates, one can seen that when UH = 1, FFA variations are greater for the highest oil temperature; but when UH = 0, this effect does not occur or is insignificant. This behavior could be explained taking into account the fact that the vapor pressure of fatty acids increases as temperature increases, and hence the fatty acid concentration in the distillates also increases. When UH = 0, the temperature in the gas distil-

TABLE 2
Free Fatty Acids Content vs. Time, Without Gas Distillates Heating (UH = 0)[a]

Additive	T(ºC)	m_{N_2} (L/h)	Assay[b]	*t* initial	*t* = 0	*t* = 1	*t* = 2	*t* = 3	*t* = 4	*t* = 5	*t* = 6	*t* = 7
Oleic acid	245	18	E9	2.38	*2.10*	*1.79*	*1.64*	*1.50*	*1.44*	*1.32*	*1.34*	—
		30	E10a	2.39	*2.36*	2.02	*1.85*	*1.65*	*1.43*	*1.25*	*1.12*	
			E10b	1.71	1.44	*1.26*	*0.91*	*0.77*	*0.62*	*0.48*	*0.44*	*0.47*
		47	E11	2.52	*2.51*	*1.87*	*1.66*	*1.33*	*1.15*	*0.90*	*0.77*	*0.68*
	265	18	E12	2.47	*2.17*	*1.97*	*1.87*	*1.82*	*1.67*	*1.58*	*1.35*	
		30	E13	2.32	*2.14*	*1.89*	*1.74*	*1.68*	*1.58*	*1.40*	*1.41*	
		47	E14	2.29	2.07	*1.25*	*0.99*	*0.82*	*0.74*	*0.70*	*0.60*	*0.55*
Olive oil distillates	245	18	E15	2.54	2.43	*1.89*	*1.68*	*1.47*	*1.39*	*1.21*	*1.02*	
	265	18	E16	2.59	2.01	*1.63*	*1.44*	*1.31*	*1.26*	*1.18*	*1.32*	

[a] The data that correspond to the steadiest values in all the measured variables have been written in italics and have been used to calculate free fatty acid distillation rate and vaporization efficiency of free fatty acids. See Table 1 for abbreviations.
[b] Assay denomination. When a letter is included, it indicates that this assay has been repeated.

TABLE 3
Comparative Results for Mixtures of Sunflower Oil[a]

Assay		T	m_{N_2}					
UH = 1	UH = 0	(°C)	(L/h)	$FFADR_{UH = 1}$	$FFADR_{UH = 0}$	$FFAC_{UH = 1}$ (%)	$FFAC_{UH = 0}$ (%)	$E_{UH = 1}/E_{UH = 0}$
Sunflower oil and oleic acid								
E1	E9	245	18	0.289*	0.080	93*	70	3.12*
E2	E10		30	0.241	0.154*	93	92*	1.88*
E3	E11		47	0.489	0.192	94	90	2.05
E4	E12	265	18	0.429*	0.071	91*	81	3.16*
E5	E13		30	0.532	0.070	85	90	5.39
E6	E14		47	0.414	0.129	94	93	2.57
Sunflower oil and olive oil distillates								
E7	E15	245	18	0.170	0.118	82	79	1.33
E8	E16	265	18	0.209	0.048	81	75	3.88

[a] The values of the deodorization performance responses (FFADR and FFAC) and the E ratios are compared for equal oil temperatures and nitrogen flow rates with respect to the heating procedure (UH = 0 and UH = 1). m_{N_2}, nitrogen flow rates; FFADR, free fatty acid distallation rate; FFAC, free fatty acid content; E, vaporization efficiency of free fatty acid. See Tables 1 and 2 for other abbreviations. *Average of two assays.

lates is lower than in the oil and there are only small changes in the differences in temperature between the oil and the gas distillates for the assayed oil temperatures. This behavior could be explained by the low thermal conductivity of the gas distillates.

Taking each table separately, for the same temperature and different nitrogen flow rates, it seems that the greater the flow rate, the greater is the variation in FFA. The cause could be that as the gas velocity increases, the drag force increases as well, and the refluxing of the distillate to the oil becomes more difficult.

In Table 3, the values of the deodorization performance responses (FFADR and FFAC) and the efficiency ratios are compared for equal oil temperatures and nitrogen flow rates with respect to the heating procedure (UH = 1 and UH = 0). The first and the second columns give the assay denomination for UH = 1 and UH = 0, respectively. The values obtained in repeated assays have been averaged out.

For the mixture of refined sunflower seed oil and oleic acid, a positive influence of the heating procedure is observed with respect to FFADR and efficiency ratios for all the nitrogen flow rates and oil temperatures assayed. The values of FFADR are at least duplicated when UH = 1, and the efficiency ratio is multiplied twice, three, four, or five times. The effects of the oil temperature and nitrogen flow rates are not clear. For the deodorization of the mixture of refined sunflower and olive oil distillates, the positive effects of heating the upper part of the deodorizer can also clearly be appreciated.

The values of FFAC are very high for all assay conditions, reaching values of 93% for the assays with the mixture of refined sunflower seed oil and oleic acid. This result implies that oil losses are very small. Significant differences were not found in the results on changing the heating procedure.

It is possible to confirm the aforementioned observations by establishing the corresponding confidence coefficients. To do this, the statistical technique of blocking with paired comparisons (9) was applied to the mixture of refined sunflower seed oil and oleic acid. The factors to be studied were heating procedure (UH = 1 and UH = 0), temperature of the oil (245 and 265°C), and nitrogen flow rate (m_{N_2} = 18 and 30 L/h; m_{N_2} = 18 and 47 L/h; and m_{N_2} = 30 and 47 L/h). The responses were FFADR, E, and FFAC. In order to ensure that the application of this technique was appropriate, the non-iteration between factors was verified using 2^3 factorial designs (three factors and two levels) (9), though the results are not presented in this paper.

The response differences (d_i) were obtained for each factor and equal conditions in the others. Subsequently, the average difference, $d_{avg} = \Sigma d_i/n$ (where n was the number of data), and the sample standard deviation $S_d = \sqrt{\Sigma(d_i - d_{avg})^2/(n - 1)}$ for each factor were calculated. The n differences of each factor (d_i) were assumed as samples of an approximately normal population of mean equal to zero, and therefore $d_{avg}/S_d/\sqrt{n}$ follows a Student t distribution with (n − 1) degrees of freedom (t_{n-1}). One-sided or two-sided significance tests were used to obtain the critical significance level α_{ctn-1} and the critical confidence coefficient, $1 - \alpha_{ctn-1}$.

Table 4 shows the block formation for UH = 1 and UH = 0, for equal conditions of oil temperature and nitrogen flow rate, along with the response differences. In the lower part of the table are the values of d_{avg}, S_d, and t_{n-1} for all the responses along with the corresponding critical confidence coefficients for the heating procedure factor. It can be appreciated that when the gas distillates are heated, FFADR and E are improved and this can be said with a confidence coefficient of 99.9%. In the same way, it can also be stated with a confidence coefficient of 88.8% that the FFAC in the recovered distillates increases.

Table 5 shows block formation for the oil temperature factor along with the response differences. In the lower part of the table, the critical confidence coefficients for all the responses are presented. E decreases for the highest oil temperature, and this can be stated with a confidence coefficient of 99.4%. The confidence coefficients for the FFDR and for the FFAC in the recovered distillates are very low, so it cannot be confirmed that the oil temperature significantly affects these responses.

Table 6 shows the blocks formation along with the response

TABLE 4
Analysis of the Heating Procedure Factor

		Block formation[a]			
Response	T(°C)	m_{N_2} (L/h)	UH = 1	UH = 0	d_i
FFADR	245	18	0.289*	0.080	0.209
		30	0.241	0.154*	0.088
		47	0.489	0.192	0.297
	265	18	0.429*	0.071	0.358
		30	0.532	0.070	0.462
		47	0.414	0.129	0.285
E	245	18	0.242*	0.078	0.164
		30	0.157	0.084	0.073
		47	0.178	0.087	0.091
	265	18	0.147*	0.046	0.101
		30	0.153	0.028	0.125
		47	0.070	0.027	0.043
FFAC	245	18	92.9*	69.5	23.4
		30	92.7	91.7*	1.0
		47	93.3	90.2	3.1
	265	18	91.7*	81.3	10.4
		30	85.2	89.9	−4.7
		47	94.0	93.3	0.7

	Critical confidence coefficients[b]			
Response	d_{avg}	S_d	t_{n-1}	$(1 - \alpha_{ctn-1})100$
FFADR	0.283	0.128	5.4241	99.9
E	0.099	0.042	5.8000	99.9
FFAC	5.630	9.950	1.3866	88.8

[a] Oil temperature and nitrogen flow rate were equal for UH = 0 and UH = 1. d_i, response differences. See Tables 1, 2, and 3 for other abbreviations. *Average of two assays.

[b] d_{avg}, average difference; S_d, standard deviation of sample; t_{n-1}, degrees of freedom; $(1 - \alpha_{ctn-1})100$, critical confidence coefficient.

TABLE 5
Analysis of the Oil Temperature Factor

		Block formation[a]			
Response	T(°C)	m_{N_2} (L/h)	T = 245°C	T = 265°C	d_i
FFADR	1	18	0.289*	0.429*	0.141
		30	0.241	0.532	0.291
		47	0.489	0.414	−0.075
	0	18	0.080	0.071	−0.009
		30	0.150*	0.070	−0.080
		47	0.192	0.129	−0.063
E	1	18	0.242	0.147*	−0.095
		30	0.157	0.153	−0.004
		47	0.178	0.070	−0.109
	0	18	0.078	0.046	−0.031
		30	0.084	0.028	−0.056
		47	0.087	0.027	−0.060
FFAC	1	18	92.9*	91.7*	−1.2
		30	92.7	85.1	−7.5
		47	93.3	94.0	0.7
	0	18	69.5	81.3	11.8
		30	91.7*	89.9	−1.8
		47	90.2	93.3	3.1

	Critical confidence coefficients[b]			
Response	d_{avg}	S_d	t_{n-1}	$(1 - \alpha_{ctn-1})100$
FFADR	0.034	0.151	0.5515	39.5
E	−0.059	0.039	−3.7056	99.3
FFAC	0.850	6.418	0.3244	24.1

[a] See Tables 1–4 for abbreviations.

TABLE 6
Analysis of the Nitrogen Flow Rate Factor[a]

	Block formation							
Response	T(°C)	UH	m_{N_2} =18 L/h	m_{N_2} =30 L/h	m_{N_2} =47 L/h	$d_{i\,(30-18)}$	$d_{i\,(47-30)}$	$d_{i\,(47-18)}$
	245	1	0.289*	0.241	0.489	−0.048	0.248	0.200
FFADR		0	0.080	0.150*	0.192	0.070	0.042	0.112
	265	1	0.429*	0.532	0.414	0.103	−0.118	−0.015
		0	0.071	0.070	0.129	−0.001	0.059	0.058
	245	1	0.242*	0.157	0.178	−0.085	0.021	−0.064
E		0	0.078	0.084*	0.087	0.006	0.003	0.009
	265	1	0.147*	0.153	0.070	0.006	−0.083	−0.077
		0	0.046	0.028	0.027	−0.018	−0.001	−0.019
	245	1	92.9*	92.7	93.3	−0.2	0.6	0.4
FFAC		0	69.5	91.7*	90.2	22.2	−1.5	20.7
	265	1	91.7	85.2	94.0	−6.5	8.8	2.3
		0	81.3	89.9	93.3	8.6	9.4	18.0

Critical confidence coefficients

	(30–18)				(47–30)				(47–18)			
Response	d_{avg}	S_d	t_{n-1}	$(1-\alpha_{ctn-1})100$	d_{avg}	S_d	t_{n-1}	$(1-\alpha_{ctn-1})100$	d_{avg}	S_d	t_{n-1}	$(1-\alpha_{ctn-1})100$
FFADR	0.031	0.068	0.9122	51.1	0.058	0.150	0.7700	50.3	0.089	0.091	1.9514	85.4
E	−0.023	0.043	−1.0698	63.7	−0.015	0.046	−0.6522	43.9	−0.038	0.040	−1.9000	84.6
FFAC	6.050	12.410	0.9750	59.9	4.330	5.585	1.5506	78.1	10.380	10.451	1.9868	85.9

[a] See Tables 1–4 for abbreviations.

differences for three associations of nitrogen flow rates: (30–18), (47–30) and (47–18). In the lower part of the same table, the resulting critical confidence coefficients for all the associations are shown. For the group 47–18 , it can be appreciated that when the nitrogen flow rate increases, FFDR, E, and the FFAC in the recovered distillates also increase, and the confidence coefficient is around 85% for all the responses. With respect to the groups 30–18 and 47–30, the confidence coefficients for all the responses are very low, and it cannot be affirmed that the flow rate of nitrogen injected was a significant influence.

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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. 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).
3. Ruíz-Méndez, M.V., A. Garrido-Fernández, F.C. Rodríguez Berbel, and E. Graciani, Relationships Among the Variables Involved in the Physical Refining of Olive Oil Using Nitrogen as Stripping Gas, *Fett/Lipid 98*:121–125 (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. Rodríguez, and M.V. Ruíz-Méndez, Physical Refining of Edible Oils Using Nitrogen as Stripping Gas. Process Optimization, *Grasas Aceites 45*:132–146 (1994).
6. Deffense, E., Refinación con Vapor: Comparación de la Teoría con la Práctica, *Dossier Oleo*:49–57 (1994).
7. Vian, A., and J. Ocón, in *Elementos de Ingeniería Química*, Aguilar, Madrid, 1964, p. 657.
8. *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.
9. Box, G.E.P., W.G. Hunter, and J.S. Hunter, in *Statistics for Experimenters. An Introduction to Design, Data Analysis, and Model Building,* John Wiley & Sons, Inc., New York, 1978, pp. 93–106, 306–374.

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