Nitrogen Bubble Refining of Sunflower Oil in Shallow Pools

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ABSTRACT: High-temperature steam deodorization of sunflower oil results in the formation of unwanted by-products, such as *trans* isomers and polymers, and partial destruction of vitamins. There is an urgent need to develop a process that replaces steam with an inert gas such as nitrogen. The use of nitrogen bubble sparging at low temperatures has recently been reported as a technique to strip volatiles from edible oils. In this study, a hypothesis was proposed that nitrogen bubbles sparged at temperatures of 25 to 150°C are able to remove odoriferous, surface-active, or volatile contaminants from shallow pools of sunflower oil. Analysis of the composition of sunflower oil that had been sparged at 3 mbar pressure showed that both the odor and peroxide content of the oil were considerably reduced to values that are commercially acceptable. Odor improvement occurred at temperatures between 100 and 150°C, while the peroxide content reduction was achieved at a temperature of 150°C. There were no significant improvements in the free fatty acid concentration or color.

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Edible oil physical refining involves degumming, bleaching, dewaxing or winterization, and deodorization/deacidification. Deodorization is an important step in edible oil refining aimed at the removal of odoriferous and taste-bearing compounds from the oil. High temperatures cause the breakdown of molecules; the combination of high temperature and low (subatmospheric) pressure aids in volatilization of molecules, which are then stripped mainly by absorption into steam bubbles (1,2). Despite the high efficiency of the current process, it is generally accepted that it is beneficial to process edible oils at low temperatures in order to prevent undesirable effects, such as partial destruction of vitamins and formation of unwanted by-products such as *trans* isomers and polymers (1,3–5). This requires replacing high-temperature steam by another inert gas such as nitrogen. Until recently, nitrogen gas has been used for deaeration during oil storage and for transportation as a nitrogen blanket to protect oil against oxidation (6–8). It has also been recommended for use as a cooling medium in the heat recovery section of the deodorization unit (9). Stripping by nitrogen gas at high temperatures was found to produce oils with a quality similar to those resulting from steam deodorization. Cheng (9) used nitrogen gas for deodorization at 230°C with a pressure of 1.3 mbar and 3.4 m^3/ton of oil. Krishnamaurthy *et al.* (10) recommended the use of a jacketed vessel with height/diameter ratios of 3–40, packing surface areas of 98–1970 m^2/m^3, operating temperatures of 160–280°C, operating pressures of 69–690 mbar, pure nitrogen gas flow rates of 0.4–9.4 m^3/h, countercurrent oil flows at 1–12 kg/h, and residence times ranging from 5 min to 2 h for deodorization of bleached soybean oil. Cvengros (11) carried out deodorization with nitrogen in a vacuum-operated thin film evaporator at 200–250°C and pressures lower than 1 mbar. Graciani Constante *et al.* (12) optimized the conditions for efficient nitrogen deodorization at operating temperature of 250–260°C, pressure of 4.5–8 mbar, nitrogen gas flow rates of 1.4–2.3 m^3/ton of oil·h, oil level at 400–500 mm, and residence times of 1.5–4 h. Ruiz-Mendez *et al.* (13) compared the performance of steam and nitrogen in removing free fatty acids from sunflower oil using a glass frit with 1 μm pore size as the sparging system. At low temperatures, nitrogen successfully removed pentane from sunflower oil (14). However, work has not been carried out to examine the extent of removal of the actual contaminant molecules from edible oils by nitrogen at low temperatures.

The use of nitrogen bubbles as a stripping medium for removal of oil contaminants at low temperatures, i.e., lower than 150°C, requires that the bubbles act in such a manner that adsorption occurs at the bubble surface in order to remove surface-active contaminants, and volatilization occurs in the bubble cavity for removal of highly volatile contaminants. In the present article, the analytical results on the composition of sunflower oil sparged with nitrogen gas at subatmospheric pressure and temperatures in the range of 25 to 150°C are critically displayed. The work presented in this paper is part of a study that was initiated to examine the potential of nitrogen bubbles to remove contaminants from edible oils at temperatures lower than those currently used in steam deodorization (i.e., below 200°C).

EXPERIMENTAL PROCEDURES

Materials. Semiprocessed (degummed, bleached, and filtered) sunflower oil was supplied by Elais S.A. (Athens, Greece). The oil was stored at 0°C in 1-L plastic containers with a headspace of nitrogen gas to prevent oxidation. Nitro-

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gen gas (99.999% purity) used for sparging during oil processing was supplied by Linde Hellas (Athens, Greece). A phosphate-free detergent (Neodisher-FLA, Dr Weighert Chemische Fabrik GmbH & Co., Hamburg, Germany) was used for cleaning glass equipment. Vacuum pump oil (Bartran 32) was purchased from BP Hellas Ltd. (Athens, Greece). Silicone heating oil (Rhodorsil 47V 350 by Rhône-Poulenc Specialties Chimiques, Marseilles, France) was used for heat transfer into the bubble column for high temperature (100 and 150°C) studies. Ethanol (96%) and commercial-grade petroleum ether (Moscholios Chemicals, Athens, Greece); sodium hydroxide, potassium iodide, phenolphthalein indicator, analytical-grade glacial acetic acid, analytical-grade chloroform, analytical-grade iso-octane, solid sodium thiosulfate, and matrix modifier lanthanum solution (0.5% wt/vol) (Merck, Darmstadt, Germany); analytical grade diethyl ether (Riedel-de Haen, Seelze, Germany); lecithin Bolec Z gel with a 2% phosphorus content (Unilever URL, Vlaardingen, The Netherlands) were all provided by Elais S.A. Phosphorus-free sunflower oil was prepared by Elais S.A. Nitrogen gas (98% purity), and argon gas used for analytical purposes were supplied by Air Liquide Hellas (Athens, Greece) and Linde Hellas, respectively. A range of Greek and European commercial sunflower oils (A–J) that were steam-deodorized were from the shelves of a food store.

Apparatus and refining procedure. The experimental setup is shown in Figure 1. The custom-designed bubble column was a 35-cm tall (oil height-to-diameter ratio of 1) cylindrical Pyrex glass vessel with a G-3 (20–30 μm) porosity sintered sparger fitted to the whole base area (construction by George Louvaris, Athens, Greece). After the insertion of an O-ring between the column top and the column cover, the cover was held in position with a stainless steel tightening ring (126–155 mm diameter; Norma 143, DN100). A 500-W heating coil immersed in the heating bath was used to heat the bubble column contents to the desired temperature. An oil ring vacuum pump (RB 4D; DV.P. Vacuum Technology s.l.r., Bolognia, Italy) was used to create and maintain subatmospheric pressures in the vessel during vacuum operation. Two 250-mL traps connected in the vacuum line between the vessel and the vacuum pump were intended to collect carryover from the bubble column in case of operational problems (e.g., foaming) and condensed volatiles produced during vacuum operation, respectively. The second trap, containing glass beads to increase the contact area and residence time of the volatiles, was maintained in a double-walled 318 stainless steel ice bath that was held at 0°C.

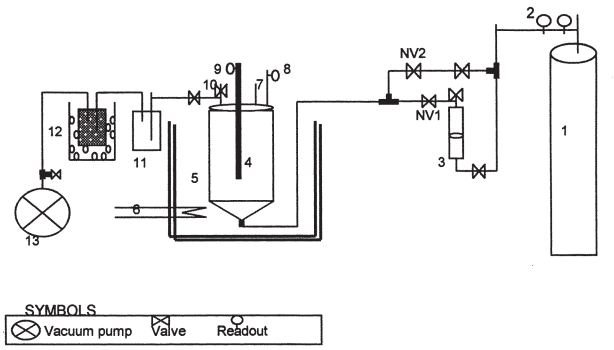

FIG. 1. Custom-designed rig for sunflower oil refining using nitrogen bubbles, at subatmospheric pressures. 1, gas cylinder; 2, manometers; 3, gas flowmeter; 4, bubble column; 5, heating bath; 6, heating coil; 7, sampling/oil introduction port; 8, pressure gauge; 9, temperature sensor; 10, connection to vacuum pump; 11, oil trap; 12, volatiles trap; 13, vacuum pump.

Nitrogen gas was introduced into the bubble column at 100 cm^3/min. The bubble column was carefully filled with 1 L of preheated sunflower oil and sparged with nitrogen gas for 4 h at temperatures of 25, 50, 100, and 150°C. For operation at temperatures of 100 and 150°C, the heating bath was filled with silicone oil (Rhodorsil 47V 350 by Rhône-Poulenc Specialties Chimiques). The column operated in two modes: (i) one-stage operation under vacuum (called vacuum operation) and (ii) two-stage operation, i.e., a sequential use of atmospheric pressure and vacuum (called combined operation). During vacuum operation, the pressure was maintained at 3 mbar. During combined operation, the oil was sparged with nitrogen gas first at atmospheric pressure for 1 h, and then under vacuum at 3 mbar for 3 h. Pressure regulation was carried out by fine tuning of a needle valve (NV1) placed between the flowmeter and the column. After 4 h of sparging in one-stage or two-stage operation, three 10-mL samples of the oil were taken in order to be analyzed. Prior to discharge of the oil at the end of each run, 200 mL of the oil was collected in a 250-mL plastic sample vial, which was then sealed, labeled, covered with aluminum foil, and stored at 0°C. These samples were used for organoleptic evaluation.

Analytical determinations. Nitrogen-sparged sunflower oil was analyzed by titration for free fatty acid and peroxide contents, spectrophotometry for color, and atomic absorption spectroscopy for phosphorus. The analyses were carried out at Elais S.A. Odor of the sunflower oil was assessed organoleptically. The range of Greek and European commercial, steam-deodorized sunflower oils was analyzed in a similar manner for comparison.

Acidity determination was by a European Economic Community (EEC) recommended method (15). Three to 5 g of oil sample was accurately weighed into a 250-mL conical flask and approximately 70 mL of 1:1 diethyl ether/ethanol (96%) solvent solution was added for dissolution and clarification of the oil. The oil sample was then titrated with 0.1 N aqueous sodium hydroxide solution using phenolphthalein indicator (1% wt/vol in 96% ethanol) until the color of the diluted oil sample turned to a permanent light pink. The amount of the added alkali was recorded. Free fatty acid content or acidity of the oil sample was expressed as percentage by weight of oleic acid using the equation, acidity = weight % of oleic acid = 2.82 (V/w), where V is the volume of alkali added (mL) and w is the oil sample weight (g).

Peroxide value (PV) determination was by an EEC recommended method (16). The weighed oil sample was dissolved

in 30 mL of acetic acid/chloroform solution (3:2 vol/vol), and 1 mL of saturated potassium iodide solution was added. The mixture was allowed to stand for exactly 1 min, and 30 mL of deionized water was added. The liberated iodine was titrated against 0.01 N sodium thiosulfate with simultaneous agitation. A Pt electrode (Metrohm 0–80°C Pt Titrode; Metrohm, Herisau, Switzerland) was used to detect the titration equivalence or end point. When not in use, the electrode was kept immersed in a cleaning solution of commercial-grade ethanol/deionized water mixture (1:1 vol/vol). PV (meq/kg oil) was calculated by the equation $PV = (V_s N \cdot 1000)/w$, where V_s is the volume of sodium thiosulfate solution used (mL), N is the normality of the same solution (meq/kg oil), and w is the oil sample weight (g).

Color evaluation was by a spectrophotometer (Photovolt model 401; Photovolt Corporation, Indianapolis, IN) that measured transmittance in a test solution on a 0–100% scale. An in-house method developed by Elais, S.A. was used, which is based on determination of the transmittance values at 430 nm using a blue color filter. The equipment was calibrated using distilled water.

Total phosphorous content of oil samples was determined in a graphite furnace atomic absorption spectrometer (Zeeman 3030 System; Perkin-Elmer, Uberlingen, Germany). Standard solutions with 10, 20, and 40 ppm phosphorus were prepared from the lecithin gel using phosphorus-free sunflower oil (0 ppm) for dilution. The equipment corrected the measured absorbance values by subtracting the interferences. The phosphorus content of the oil samples was evaluated from the corrected absorbance values against the standard calibration curves that were obtained prior to the analysis of the oil samples.

Organoleptic evaluation of odor was by a panel consisting of 14 members. The methodology was based on that described in the Official EEC Methods of Analysis (17). Each panelist recorded the odor in the supplied questionnaire. The final odor score for each sample was calculated as the arithmetic mean of the scores given by the panelists.

Reproducibility of analytical experiments was assessed by one-way and two-way analysis of variance (ANOVA) at a significance level of 1%. The effect of operating parameters on properties of sunflower oil was examined by one-way and two-way ANOVA (5% level of significance), *t*-tests, and regression analysis.

RESULTS

The analytical results for pretreated, nitrogen-sparged, and commercial sunflower oils are displayed in Table 1. The free fatty acid content of nitrogen-sparged oils at temperatures in the range of 25 to 150°C was similar to that of the pretreated sunflower oil (P = probability value = 0.09). Although there was significant variation among the commercial, steam-deodorized oils ($P < 0.01$), these had significantly lower free fatty acid contents than the nitrogen-sparged oils ($P < 0.01$). It is clear that nitrogen sparging needs to be carried out at

TABLE 1
Analytical Results for Nitrogen-Sparged and Commercial Sunflower Oils[a,b]

Sample	Free fatty acid content (% acidity)		Peroxide value (meq/kg oil)		Color (% light transmission)		Phosphorus concentration (ppm)		Odor score (–)	
	Mean	SD	Mean	SD	Mean	SD	Mean	SD	Mean	SD
Pretreated sunflower oil										
25°C	0.80	0.03	15.13	0.57	45.47	0.64	4.58	0.68	5	0.00
Vacuum-treated sunflower oil										
25°C	0.80	0.03	10.30	0.22	43.43	0.96	4.32	0.72	6	0.22
50°C	0.81	0.01	10.12	0.18	45.83	0.89	4.04	1.33	6	0.48
100°C	0.79	0.00	13.10	0.41	53.17	1.22	4.32	1.01	8	0.27
150°C	0.84	0.01	1.40	0.33	32.00	1.40	3.23	0.72	8	0.16
Atmospheric pressure/vacuum-treated sunflower oil										
25°C	0.81	0.02	9.91	0.25	47.07	1.89	5.45	0.65	5	0.33
50°C	0.83	0.02	8.78	0.17	51.17	1.22	6.64	1.14	6	0.47
100°C	0.84	0.02	11.49	0.59	49.33	1.56	6.44	0.92	8	0.58
150°C	0.83	0.00	1.72	0.91	34.97	0.82	7.37	0.40	8	0.05
Steam-deodorized finished products (at temperatures >200°C)										
A	0.15	0.00	1.80	0.29	74.37	0.78	1.17	0.11	9	0.60
B	0.12	0.01	1.44	0.31	84.17	1.24	1.13	0.18	9	0.81
C	0.14	0.01	0.40	0.10	72.73	0.78	1.10	0.07	9	0.50
D	0.11	0.00	5.57	0.54	75.10	0.33	N/A	N/A	9	0.73
E	0.16	0.00	0.91	0.04	70.07	0.31	1.10	0.13	9	0.68
F	0.10	0.00	3.82	0.15	78.70	0.33	1.50	0.07	9	0.66
G	0.29	0.01	N/A	N/A	60.70	0.33	N/A	N/A	9	0.60
H	0.24	0.01	2.99	0.16	55.20	0.40	1.23	0.11	N/A	N/A
J	0.13	0.00	3.18	0.23	65.90	0.33	N/A	N/A	N/A	N/A

[a]Specifications: Free fatty acid content <0.10%; peroxide value = 0 meq/kg oil; Phosphorus concentration <5 ppm (Elais, S.A., Athens, Greece, 1998).
[b]Abbreviations: SD, standard deviation; N/A, values are not available.

temperatures in excess of 150°C in order to remove free fatty acids.

The peroxide content of the pretreated, semiprocessed sunflower oil was higher than that of the seven selected commercial oils. The variation in the peroxide content among the commercial, steam-deodorized oils was significant ($P < 0.01$). Although the observed lowering in the PV after nitrogen bubble sparging at temperatures in the range of 25 to 100°C was significant ($P < 0.01$) and the oil differed from the pretreated one, the oil was not commercially acceptable (Table 1). At 150°C, in both the vacuum and combined mode operated systems, rapid decreases in PV were achieved, and the peroxide content of these oils was comparable to the values obtained for commercial finished oils ($P = 0.60$). Although none of these gave a zero value as specified by Elais, S.A. (1998; T. Helmis, personal communication), the results clearly show that removal of peroxides is achievable with nitrogen bubble sparging at temperatures in excess of 100°C.

An increase in the percentage value of light transmission values implied that more light passed through the sample under investigation. There was a significant variation in color of the commercial oil samples ($P < 0.01$). The color of the sunflower oil sparged with nitrogen at temperatures in the range of 25 to 150°C was similar to that of the pretreated samples ($P = 0.82$). The color of nitrogen-sparged sunflower oils was significantly different from the color of the commercial steam-deodorized oils ($P < 0.01$).

The odor of sunflower oil samples was measured on a scale of 1–10, called the odor scale, 10 being the least odoriferous sample. During both vacuum and combined operations, the odor score values increased with temperature. At 100 and 150°C, a value of 8 ± 0.58 was achieved in both systems, showing significant success in removal of volatile odoriferous species. The odor of sunflower oil sparged with nitrogen at temperatures of 100 and 150°C was shown to be close but not equal to the odor of commercial, steam-deodorized oils with scores of 9 ± 0.81 ($P = 0.62$). The true nature of the odoriferous components removed by relatively high temperature nitrogen bubble sparging remains to be established by further analyses such as chromatography and mass spectroscopy.

DISCUSSION

Odoriferous components are volatile molecules, such as small molecular weight aldehydes and ketones, and in edible oils are usually present in concentrations of a few parts per million (2,18,19). Characterization of volatiles is possible by the use of very sensitive methodologies, both for collection and analysis. Volatile molecules identified in edible oils include pentane, pentanal, hexanal, octenal, and decadienal (1,20,21). Because volatiles are detectable organoleptically, sensory evaluation is commonly used for assessing flavor and odor of oils (11,12,22). The organoleptic panel tests used in this work showed that there was a significant improvement in the odor of sunflower oil that was sparged with nitrogen at temperatures of 100 and 150°C. A significant reduction in the amount of peroxides was also achieved by nitrogen gas sparging at 150°C. The PV of the sunflower oil sparged with nitrogen at 150°C was within the commercially acceptable values. At temperatures of about 120°C, peroxides decompose to smaller molecular weight volatile and nonvolatile molecules such as aldehydes and ketones (Elais, S.A., 1998; G. Michaelidis, personal communication). It is most likely that some of these smaller molecules were removed by volatilization into the bubbles. Therefore, nitrogen bubble sparging at 150°C can potentially be used to remove both volatile odoriferous components and peroxides from edible oils.

Evidence that removal of contaminants from sunflower oil occurred during nitrogen bubble sparging under vacuum was provided by the observation that the vacuum pump had a reduced performance at the end of high-temperature (100 and 150°C) operations. The sealant oil in the vacuum pump needed to be changed between runs in order to achieve a pressure of 3 mbar. Contamination of vacuum sealant oils with volatile components is a well-known, major problem in vacuum engineering (23). The strong, unpleasant odor that the sealant oil possessed during nitrogen bubble sparging was further evidence that components from sunflower oil had been removed and subsequently carried into the vacuum pump oil. The odor was also noticeable at the lower temperatures of 25 and 50°C; however, it was significantly milder. The retention of odoriferous species in the sealant oil suggested that these molecules were soluble in the sealant oil. The implementation of sophisticated trap designs for the collection and condensation of volatiles is recommended in order to minimize carryover of odoriferous compounds into the vacuum pump.

Nitrogen gas sparging did not affect the free fatty acid, phosphatide, or carotene content of sunflower oil. The lack of change of color during nitrogen bubble sparging was also reported in oils sparged with nitrogen at temperatures as high as 250–260°C (12). Color components such as carotenes are broken down at high temperatures during steam deodorization (1,24). Further reduction in color can be achieved by hydrogenation (Elais, S.A., 1998; Mr. G. Michaelidis, personal communication). Free fatty acids and phosphatides may have been adsorbed onto bubble interfaces. However, an absence of notable changes in the composition of sunflower oil with respect to these components after sparging at temperatures of up to 150°C suggests that these were neither adsorbed nor volatilized. Similar temperatures to those used during steam deodorization (in excess of 170°C) are necessary for their removal (25–27). Rivers (28) showed that undesirable components can be removed in a series of isothermal stations in the following order: aldehydes and ketones at 140°C; tocopherols at 143°C; and free fatty acids, peroxides, and hydroperoxides at 254°C.

This work shows that at defined operating conditions, some volatile and odoriferous components may be removed from shallow pools of sunflower oil by using nitrogen bubble sparging at temperatures below 150°C. Further volatilization

of high molecular weight species such as free fatty acids may be achieved by increasing the temperature to 180°C. These studies will be pursued in the future.

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