JOURNAL OF AGRICULTURAL AND FOOD CHEMISTRY

Fate of Oxidized Triglycerides during Refining of Seed Oils

TOMMASO GOMES,* FRANCESCO CAPONIO, AND DEBORA DELCURATOLO

Dipartimento di Progettazione e Gestione dei Sistemi Agro-Zootecnici e Forestali, Università degli Studi di Bari, Via Amendola 165/a, I-70126 Bari, Italy

The evolution of oxidized triglycerides (ox-TG) during industrial refining was studied in soybean, sunflower, peanut, and corn oils. The analytical techniques used were silica gel column chromatography and high-performance size exclusion chromatography. The decrease in ox-TG during refining (42.3% on average) was accompanied by an increase in triglyceride oligopolymers (TGP). The inverse correlation between the two lipid groups suggests that the decrease in ox-TG during refining was due in part to the occurrence of polymerization reactions. An inverse correlation was also found between the percentage sum of ox-TG + TGP and percent TGP, indicating that a part of the ox-TG also underwent degradation or transformation reactions. On average, almost 58% of the ox-TG remained unchanged during refining and, of the rest, about half was involved in polymerization reactions and half in degradation or transformation reactions.

KEYWORDS: HPSEC; oxidized triglycerides; refined seed oils; triglyceride oligopolymers

INTRODUCTION

It is well-known that triglyceride oligopolymers (TGP) are formed during vegetable oil refining (*1*−*3*). Many authors consider these compounds to be reliable indices of oxidative degradation due to their high stability and low volatility. TGP are generally formed during the bleaching and especially deodorization steps of refining because of the high temperatures in use. The amounts of TGP commonly found in vegetable oils are generally ∼1% with respect to oil (*4*). Information on the quantitative variations in oxidized triglycerides (ox-TG) is more limited, although ox-TG constitute another important class of oxidized lipids (*5*, *6*), which, together with TGP, concur in a better measure of the level of oxidation of refined oils (*7*). There is no general agreement concerning the fate of ox-TG during vegetable oil refining (*8*, *9*).

The analytical determination of TGP and ox-TG is carried out in two phases. In the first phase, silica gel column chromatography is used to separate the polar compounds (PCs) from the oil as prescribed by the IUPAC method (*10*). Polar compounds have a greater polarity than do unaltered triglycerides and are mainly composed of TGP, ox-TG, partial glycerides, sterols, triterpenic alcohols, and free fatty acids (FFA). The quantitative determination of these substances is performed during the second phase when the PCs are analyzed by high-performance size exclusion chromatography (HPSEC). Good separations of the various classes of substances are generally obtained, but it is difficult to achieve their quantitative determination because each chromatogram peak corresponds to a complex group of substances. The quantitative determination by HPSEC of the substances constituting the PCs has only recently been improved (*11*).

* Author to whom correspondence should be addressed (telephone +39 080 5442939; fax +39 080 5442939; e-mail tommaso.gomes@agr.uniba.it).

The aim of this paper was to study ox-TG during the refining procedures for seed oils performed at different industrial establishments by using the recently improved analytical method and to check whether the ox-TG remained unchanged or had measurable changes in their concentrations. This is important to assess the state of oxidation of oils and especially of recently refined oils.

MATERIALS AND METHODS

Samples. Two series of samples of sunflower, peanut, and soybean oils and a single series for corn oil were collected at different refining plants in southern Italy over a two-year period. Each series comprised unprocessed crude oil and the same oil after the steps of conventional refining (degumming−alkali refining, bleaching, and deodorization). The processing conditions were the ones generally used for these oils (*12*−*15*). The samples were collected in triplicate and were representative of large stocks of oils undergoing refining. The samples were kept in a freezer at −20 °C from the moment they reached the laboratory until they were analyzed.

Analytical Determinations. Each sample was thawed at room temperature and percent FFA, peroxide value (PV), specific extinction at 232 nm (K_{232}) (*16*), and *p*-anisidine value (*p*-AV) (*17*) were determined. Polar compounds were determined in each sample by silica gel column chromatography as described by the IUPAC method (*10*). Polar compounds were submitted to HPSEC to determine TGP, ox-TG, and diglycerides (DG). The chromatographic system consisted of a Perkin-Elmer series 10 pump, a model 7125 S sample injector (Rheodyne), a 50 μL injector loop, and a series of three 0.75 cm i.d. × 30 cm PL-gel columns (Perkin-Elmer Ltd., Beaconsfield, U.K.). The columns were packed with highly cross-linked styrene divinylbenzene copolymers with a particle diameter of 5 μm and pore diameters of 500, 500, and 100 Å, respectively. A 7.5 mm i.d. × 5 cm PL-gel guard column (Perkin-Elmer Ltd.) was used. The detector was a differential refractometer (Shimadzu model RID 6A, Shimadzu Corp.) connected to an integrator. The elution solvent used was CH_2Cl_2 for HPLC at a

10.1021/jf026136t CCC: $25.00
Published on Web 06/24/2003

Table 1. Accuracy and Precision of Main Substance Classes Constituting the Polar Compounds

determination	accuracy (recovery %)	precision (RSD %)
triglyceride oligopolymers	95.3–99.9	1.18–3.06
oxidized triglycerides	101.3–106.9	0.23–2.38
diglycerides	96.1–105.9	0.95–2.82

flow rate of 1.0 mL/min. The procedures for identifying the peaks on each chromatogram and for the quantitative assessment of the classes of compounds under investigation were carried out as described elsewhere (*11*, *18*). Known amounts of TGP, ox-TG, and DG were obtained by preparative gel permeation chromatography of PCs derived from a refined peanut oil and then used as standards in the HPSEC method. The amount collected from each standard, corresponding to a given class of compounds, was used to prepare a stock solution in CH_2Cl_2 from which solutions containing different concentrations were made by successive dilutions. These solutions were analyzed by HPSEC following the analytical method we developed. The calibration curves were obtained by plotting the amounts of standards (micrograms) that had been injected into the HPSEC system loop against the areas of the corresponding chromatogram peaks (*11*). To measure the accuracy and precision of the method used, known quantities of TGP, ox-TG, and DG were added in escalating amounts to a solution containing a known concentration and composition of PCs. The HPSEC determination was repeated three times for each sample. Precision was expressed as the relative standard deviation (RSD %) and accuracy as the percent amount recovered. The results obtained are shown in **Table 1**.

Statistics. Linear regression analysis was carried out, and Pearson's product−moment correlation coefficients between the classes of substances of interest were evaluated. ANOVA analysis was performed to check the occurrence of differences in each class of substances during refining.

RESULTS AND DISCUSSION

Table 2 shows the routine parameters for each oil sample, and **Table 3** shows the PC values and the results of the HPSEC analyses on the PCs that provided information on the classes of substances they contain.

The percent FFA, PV, *p*-AV, and specific extinction at 232 nm (K_{232}) of the refined oils were similar to the values generally found for these types of oils. However, these conventional analyses may not be of any help for evaluating the state of oxidation and hydrolysis of recently processed oils because fatty acids are removed and hydroperoxides are degraded or transformed during processing, whereas K_{232} and *p*-AV partly depend on the refining conditions (*19*−*21*). More reliable information may be obtained from HPSEC analysis of the PCs.

Correct separation of PCs from the oils has always been verified by TLC as prescribed by the IUPAC method. This is a critical step for the subsequent HPSEC determination because when low-polarity substances are not completely removed from the PCs, any unaltered triglycerides present are consequently evaluated as ox-TG, thus preventing the study of such classes of compounds during refining.

Table 3 shows that the PC values decreased by an average of 34% during refining (range = 28−43%). The step exhibiting the greatest reduction of PCs was generally degumming−neutralization, and this reduction was mainly due to the removal of the FFA. Because PCs include classes of oxidative and hydrolytic substances, their determination may yield an initial indication of the overall degradation of refined oils.

In some cases, the amount of DG apparently decreased during refining, whereas in other cases it remained virtually unchanged. Determination of DG in refined oils is instrumental in detecting the true level of hydrolytic degradation.

Table 2. Mean Results of the Routine Analyses for Each Oil Sample Examined[a]

sample	FFA (%)	PV (mequiv/kg)	K_{232}	*p*-AV
sunflower 1				
crude	1.16	12.0		3.77
degummed–neutralized	0.05	15.0		2.90
bleached	0.11	1.2		11.96
deodorized	0.06	0.3	2.501	5.26
sunflower 2				
crude	2.13	5.3		4.25
degummed–neutralized	0.21	6.9		5.56
bleached	0.23	2.3		10.01
deodorized	0.09	0.2	2.265	4.41
peanut 1				
crude	0.56	11.3		2.70
degummed–neutralized	0.12	13.4		2.80
bleached	0.17	0.4		10.91
deodorized	0.11	0.0	2.641	4.75
peanut 2				
crude	1.63	9.0		2.88
degummed–neutralized	0.15	6.6		6.68
bleached	0.15	0.6		5.81
deodorized	0.05	0.0	4.963	5.32
soybean 1				
crude	0.83	5.8		4.47
degummed–neutralized	0.12	6.3		5.21
bleached	0.11	0.1		9.60
deodorized	0.05	0.0	5.752	5.46
soybean 2				
crude	1.47	3.8		4.88
degummed–neutralized	0.11	5.0		5.65
bleached	0.08	0.3		9.17
deodorized	0.06	0.0	5.736	5.86
corn				
crude	1.77	2.6		7.75
degummed–neutralized	0.05	5.7		4.89
bleached	0.06	0.7		11.62
deodorized	0.05	0.0	4.046	3.48

[a] FFA, free fatty acids; PV, peroxide value; K_{232}, specific absorption at 232 nm; *p*-AV, *p*-anisidine value.

Triglyceride oligopolymers were evaluated in all of the crude oils. Triglyceride oligopolymer values did not change or changed only slightly during degumming−neutralization. The amount of TGP started to increase substantially during bleaching because of the effect of bleaching earth and then during deodorization, which is the most important heating phase of the whole refining process as indicated by our group and others (*22*, *23*). On average, the amount of TGP measured in the refined oils was 6.3 times greater than those of the crude oils (range = 3.1−13.5).

As may be observed from **Table 3**, compared to the levels measured in the crude oils the amount of ox-TG did not change significantly after the degumming−neutralization phase ($p < 0.01$) but generally declined substantially during bleaching and deodorization regardless of the industrial plant the oils came from. **Figure 1** shows, for a sunflower oil, the HPSEC chromatograms of the PCs in the crude oil, in the degummed−neutralized, bleached oil, and in the degummed−neutralized, bleached, and deodorized sunflower oil. Because the amount of oil used for the analysis was the same for all of the samples, the increase in TGP (peak 1) and the decrease in ox-TG (peak 2) during refining are directly visible. Peak 4 comprises sterols and triterpenic alcohols, as ascertained in a previous paper (*24*), and it decreases slightly during processing in agreement with the literature (*25*). Peak 5 contains the FFA and appears to be

Table 3. Percentage Values (w/w) in Oil of the Polar Compounds and of the Main Substance Classes Constituting Them for Each Oil Sample Examined[a]

sample	PCs (%)	TGP (%)	ox-TG (%)	DG (%)
sunflower 1				
crude	4.16 ± 0.13a	0.12 ± 0.01a	1.28 ± 0.03a	1.16 ± 0.03a
degummed–neutralized	2.73 ± 0.10b	0.08 ± 0.01b	1.33 ± 0.03a	1.00 ± 0.03b
bleached	2.64 ± 0.09b	0.23 ± 0.01c	1.01 ± 0.02b	1.02 ± 0.03b
deodorized	2.62 ± 0.09b	0.37 ± 0.01d	0.93 ± 0.02c	1.02 ± 0.02b
sunflower 2				
crude	6.57 ± 0.20a	0.15 ± 0.01a	2.10 ± 0.05a	1.69 ± 0.04a
degummed–neutralized	4.75 ± 0.15b	0.21 ± 0.01b	2.11 ± 0.05a	1.74 ± 0.04a
bleached	4.33 ± 0.14c	0.47 ± 0.01c	1.67 ± 0.04b	1.74 ± 0.04a
deodorized	3.75 ± 0.14d	0.80 ± 0.02d	1.09 ± 0.04c	1.58 ± 0.04b
peanut 1				
crude	6.04 ± 0.20a	0.09 ± 0.01a	3.50 ± 0.09a	1.59 ± 0.04a
degummed–neutralized	5.71 ± 0.20a	0.14 ± 0.01b	3.72 ± 0.11a	1.42 ± 0.04b
bleached	4.43 ± 0.15b	0.50 ± 0.01c	2.07 ± 0.04b	1.39 ± 0.03b
deodorized	3.92 ± 0.14c	0.60 ± 0.02d	1.85 ± 0.04b	1.08 ± 0.03c
peanut 2				
crude	10.98 ± 0.38a	0.08 ± 0.01a	5.90 ± 0.14a	3.07 ± 0.05a
degummed–neutralized	8.86 ± 0.26b	0.28 ± 0.01b	5.75 ± 0.14a	2.38 ± 0.05b
bleached	8.55 ± 0.25bc	0.31 ± 0.01b	5.64 ± 0.13a	2.18 ± 0.05c
deodorized	7.90 ± 0.25c	1.08 ± 0.03c	4.14 ± 0.09b	2.41 ± 0.05b
soybean 1				
crude	5.33 ± 0.18a	0.15 ± 0.01a	2.72 ± 0.05a	1.24 ± 0.03a
degummed–neutralized	4.52 ± 0.17b	0.14 ± 0.01a	2.64 ± 0.06a	1.24 ± 0.04a
bleached	4.29 ± 0.16b	0.31 ± 0.01b	2.26 ± 0.04b	1.25 ± 0.04a
deodorized	3.78 ± 0.12c	0.74 ± 0.02c	1.46 ± 0.03c	1.23 ± 0.03a
soybean 2				
crude	8.70 ± 0.28a	0.20 ± 0.01a	5.00 ± 0.12a	1.61 ± 0.05a
degummed–neutralized	7.30 ± 0.25b	0.28 ± 0.01b	4.95 ± 0.10a	1.59 ± 0.04a
bleached	6.54 ± 0.23c	0.61 ± 0.02c	3.88 ± 0.09b	1.63 ± 0.04a
deodorized	5.94 ± 0.21d	1.30 ± 0.03d	2.62 ± 0.06c	1.63 ± 0.05a
corn				
crude	6.76 ± 0.23a	0.15 ± 0.01a	1.77 ± 0.04a	2.48 ± 0.05a
degummed–neutralized	4.99 ± 0.19b	0.13 ± 0.01a	1.80 ± 0.03a	2.43 ± 0.05ab
bleached	4.78 ± 0.18b	0.21 ± 0.01b	1.57 ± 0.03b	2.41 ± 0.05ab
deodorized	4.18 ± 0.15c	0.65 ± 0.02c	0.89 ± 0.02c	2.32 ± 0.05b

[a] Results of statistical analysis at $p < 0.01$. Mean values ± SD; one common letter following an entry indicates no significance. PCs, polar compounds; TGP, triglyceride oligopolymers; ox-TG, oxidized triglycerides; DG, diglycerides.

smaller in degummed−neutralized and bleached oil (chromatogram B) and in degummed−neutralized, bleached, and deodorized oil (chromatogram C) than in crude oil. The average decrease in ox-TG after full refining of crude oil was 42.3% (range = 27.3−49.7%). As observed from **Figure 2**, the percent of ox-TG was linearly related to the percent amounts of TGP. The regression lines show an inverse correlation for all of the series of oils with $p < 0.01$ except for peanut oil series 1 ($p < 0.05$). The results suggested that the decrease in ox-TG during refining is caused by the occurrence of polymerization reactions.

During refining, due to the high temperatures and varying degrees of vacuum, unaltered triglycerides may undergo direct polymerization. However, the polarity of these oligopolymers is very close to that of unaltered triglycerides and clearly lower than that of the oligopolymeric triglycerides found in PCs deriving from polymerization of ox-TG (unpublished results). Therefore, in silica gel column separation, when oligopolymers of unaltered triglycerides are present, they are found among the nonpolar compounds and are thus absent in the PCs. Hence, no interference from them can be postulated.

Moreover, one may assume that, during some refining stages, the unaltered triglycerides may undergo some type of oxidation and then polymerization or degradation. During such stages it is not possible to measure the exact percentage of ox-TG, TGP, and products of degradation present at a given moment. In our experiment, samples were collected at the end of the stage considered and single classes of compounds of interest determined, and this is what constituted the final result. Finally,

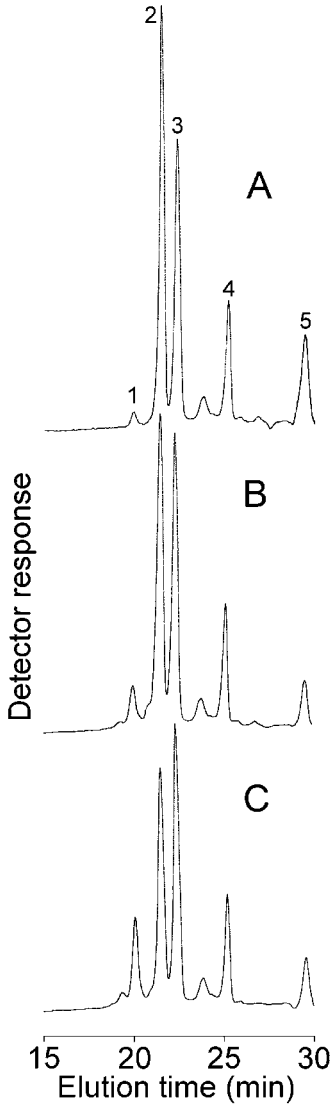

Figure 1. HPSEC analysis of polar compounds of a sunflower oil during refining: (A) crude oil; (B) degummed–neutalized and bleached oil; (C) degummed–neutalized, bleached, and deodorized oil. Peaks: (1) triglyceride oligopolymers; (2) oxidized triglycerides; (3) diglycerides; (4) sterols and triterpenic alcohols; (5) free fatty acids.

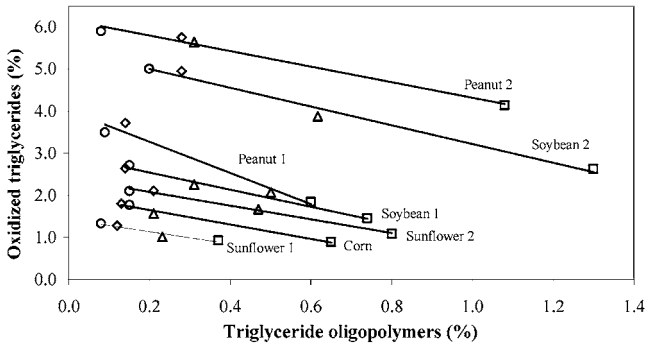

Figure 2. Percent values of ox-TG plotted against percent values of TGP during refining: (○) crude oil; (◇) degummed–neutralized oil; (△) bleached oil; (□) deodorized oil.

concentrations of the various classes of substances were expressed as weight percent in the oil and not as total amounts, so we were not interested in measuring the amounts of these substances in the oil removed with the bleached earths or in the soapy pastes and wash waters.

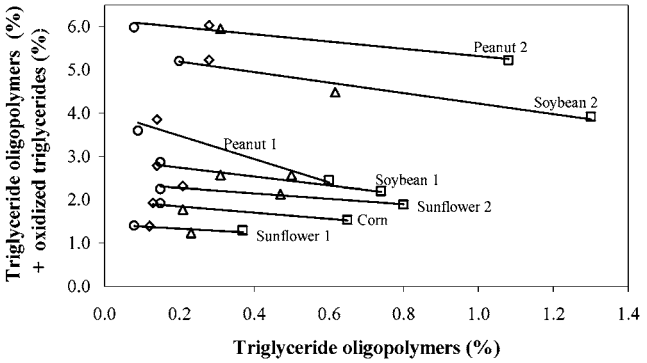

Figure 3. Percent values of ox-TG plus TGP plotted against percent values of TGP during refining: (○) crude oil; (◇) degummed–neutralized oil; (△) bleached oil; (□) deodorized oil.

A linear relationship was found between the percentage sum of TGP + ox-TG and the percent TGP. The relevant regression lines had negative slopes and showed a correlation for all of the series of samples at $p < 0.05$ (**Figure 3**). If the decrease in ox-TG were due to only polymerization reactions, the percentage sum of TGP + ox-TG would not diminish when TPG increased because a decrease in weight of the ox-TG would bring about an increase in weight of the TGP, thus tracing a straight line parallel to the abscissa. The fact that the percentage sum of TGP + ox-TG decreases as the percent amount of TGP increases may be explained by assuming that not all of the ox-TG was involved in polymerization reactions but rather some—especially the hydroperoxides—were degraded during refining, yielding lower molecular weight substances that were mainly removed during deodorization. Artifacts might also have formed that were partly absorbed by bleaching earths. This is in agreement with what is currently known about the refining of vegetable oils (*25*). We were able to quantify the percent ox-TG that had been degraded or transformed, that underwent polymerization, or that remained unchanged during refining. The percent amount (w/w) of ox-TG that remain unaltered was calculated as the ratio of ox-TG of refined oil to that of crude oil multiplied by 100:

$$\text{percentage of unchanged ox-TG} = [\text{ox-TG \% (refined oil)}]/[\text{ox-TG \% (crude oil)}] \times 100$$

The percent amount (w/w) of ox-TG that underwent polymerization reactions was computed as the ratio between the increase in TGP in refined oil over the initial TGP in crude oil, and the ox-TG in crude oil, again multiplied by 100.

$$\text{percentage of polymerized ox-TG} = [\text{TGP \% (refined oil)} - \text{TGP \% (crude oil)}]/[\text{ox-TG \% (crude oil)}] \times 100$$

The percent amount (w/w) of ox-TG that were degraded and/or transformed was derived by subtracting the percent unaltered ox-TG and percent polymerized ox-TG from 100%.

The results obtained by applying the formulas indicated above on all of the series of refined oils are shown in **Table 4**. On average, ~58% of ox-TG remained unaltered in the oil during processing. Of the rest, about half was involved in polymerization reactions and the other half in degradation reactions. The results vary greatly because they refer to the refining of different oils under different conditions. However, **Table 4** offers readers a glimpse of the mean percent amounts of ox-TG that were unaltered, polymerized, and degraded and/or transformed at the end of the refining process.

Table 4. Fate of the Oxidized Triglycerides at the End of the Refining Process of Seed Oils (Percent Values with Respect to Crude Oil)

oil	degraded or transformed (%)	polymerized (%)	unmodified (%)
sunflower 1	7.81	19.53	72.66
sunflower 2	17.14	30.95	51.91
peanut 1	32.57	14.57	52.86
peanut 2	12.88	16.95	70.17
soybean 1	24.63	21.69	53.68
soybean 2	25.60	22.00	52.40
corn	21.47	28.25	50.28
mean value	20.30	21.99	57.71
SD	8.36	5.86	9.45
min value	7.81	14.57	50.28
max value	32.57	30.95	72.66
RSD %	41.18	26.65	16.37

Table 5. Oxidation (Percentages of Twice Triglyceride Oligopolymers + Oxidized Triglycerides) of Crude Oils and Corresponding Percent Triglyceride Oligopolymers in Deodorized Oils for the Sunflower, Peanut, and Soybean Refining Series[a]

	crude oil	deodorized oil
oil	2 TGP + ox-TG (%)	TGP (%)
sunflower 1	1.52	0.37
sunflower 2	2.40	0.80
peanut 1	3.68	0.60
peanut 2	6.04	1.08
soybean 1	3.02	0.74
soybean 2	5.40	1.30

[a] TGP, triglyceride oligopolymers; ox-TG, oxidized triglycerides.

In a previous paper (*23*) we found that an appropriate way to measure the oxidative level of crude oils in terms of ox-TG and TGP was 2 times TGP % + ox-TG %. Using this formula, the calculated levels of oxidation present in crude sunflower, peanut, and soybean oils are given in **Table 5** along with the percent amounts of TGP present in the corresponding refined oils. In the oils coming from the same source, the higher the level of oxidative degradation in the crude oil, the greater the percent amount of TGP found in the refined oil, even though the oils were processed at different industrial plants. The fact that the percent amount of TGP in the refined oil depended on the level of oxidation of the crude oil lends support to what we found in this investigation.

LITERATURE CITED

(1) Eder, S. R. The formation of artifacts during deodorization of fats and oils. *Fette Seifen Anstrichm.* **1982**, *84*, 136−141.

(2) Gomes, T.; Catalano, M. Quality characters of edible oils. Dimeric triglycerides. *Riv. Ital. Sostanze Grasse* **1988**, *65*, 125−127.

(3) Dobarganes, M. C.; Perez-Camino, M. C.; Marquez-Ruiz, G. Application of minor glyceridic component determination to the evaluation of olive oils. In *Actes du Congrès International "Chevreul" pour l'étude des Corps Gras*; Premier Congrès Eurolipid, Angers, June 6−9, 1989; ETIG: Paris, France, 1989; pp 578−584.

(4) Gomes, T. Oligopolimeri in oli vegetali sottoposti a trattamenti termici. *Riv. Ital. Sostanze Grasse* **1988**, *65*, 433−438.

(5) Perez-Camino, M. C.; Marquez-Ruiz, G.; Dobarganes, M. C. Determination cuantitativa de trigliceridos oxidatos para la evaluacion global del grado de oxidation en aceites y grasas comestibles. *Grasas Aceites* **1990**, *41*, 366−370.

(6) Hopia, A. Analysis of high molecular weight autoxidation products using high performance size exclusion chromatography: I. Changes during autoxidation. *Lebensm.-Wiss. Technol.* **1993**, *26*, 563−567.
(7) Gomes, T.; Caponio, F. Possibility to improving the quality characteristics of olive-pomace oil and enhancing its differentiation from refined olive-pomace oil. *J. Sci. Food Agric.* **2001**, *81*, 62−67.
(8) Hopia, A. Analysis of high molecular weight autoxidation products using high performance size exclusion chromatography: II. Changes during processing. *Food Sci. Technol.* **1993**, *26*, 568−571.
(9) de Greyt, W. F.; Kellens, M. J.; Huyghebaert, A. D. Polymeric and oxidized triglyceride content of crude and refined vegetable oils−An overview. *Fett/Lipid* **1997**, *99*, 287−290.
(10) IUPAC. *Standard Methods for the Analysis of Oils, Fats and Derivatives*, 7th ed.; Blackwell Scientific Publications: Oxford, U.K., 1987; No. 2.507.
(11) Gomes, T.; Caponio, F. Effort to improve the quantitative determination of oxidation and hydrolysis compound classes in edible vegetable oils. *J. Chromatogr. A* **1999**, *844*, 77−86.
(12) Devidson, H. F.; Campbell, E. J.; Bell, R. J.; Pritchard, R. A. Sunflower oil. In *Bailey's Industrial Oil and Fat Products*, 5th ed.; Hui, Y. H., Ed.; Wiley: New York, 1996; Vol. 2, pp 603−689.
(13) Young, C. T. Peanut oil. In *Bailey's Industrial Oil and Fat Products*, 5th ed.; Hui, Y. H., Ed.; Wiley: New York, 1996; Vol. 2, pp 377−392.
(14) Sipos, E. F.; Szuhaj, B. F. Soybean oil. In *Bailey's Industrial Oil and Fat Products*, 5th ed.; Hui, Y. H., Ed.; Wiley: New York, 1996; Vol. 2, pp 497−601.
(15) Strecker, L. R.; Bieber, M. A.; Maza, A.; Grossberger, T.; Dokoczynski, W. J. Corn oil. In *Bailey's Industrial Oil and Fat Products*, 5th ed.; Hui, Y. H., Ed.; Wiley: New York, 1996; Vol. 2, pp 125−158.
(16) *Off. J. Eur. Communities* **1991**, *248* (Sept 5, 1991), EC Regulation 2568/91.
(17) IUPAC. *Standard Methods for the Analysis of Oils, Fats and Derivatives*, 7th ed.; Blackwell Scientific Publications: Oxford, U.K., 1987; No. 2.504.
(18) Gomes, T. Oligopolymer, diglyceride and oxidized triglyceride contents as measure of olive oil quality. *J. Am. Oil Chem. Soc.* **1992**, *69*, 1219−1223.
(19) Amati, A.; Mincuzzi, A.; Losi, G. Sulla decolorazione degli oli di oliva. Nota I: Variazioni di alcune caratteristiche chimico-fisiche in funzione delle condizioni operative. *Riv. Ital. Sostanze Grasse* **1969**, *46*, 73−79.
(20) Tiscornia, E.; Forina, M.; Evangelisti, F. Composizione chimica dell'olio di oliva e sue variazioni indotte dal processo di raffinazione. *Riv. Ital. Sostanze Grasse* **1982**, *59*, 519−556.
(21) Hastert, R. C. Adsorptive treatment of edible oils. In *Introduction to Fats and Oils Techonology*; Wan, P. J., Ed.; AOCS: Champaign, IL, 1992; pp 94−105.
(22) Perez-Camino, M. C.; García, J. M.; Castellano, J. M. Polar compound concentrations in virgin oils from stored cultivar *Picual* olive fruits. *J. Agric. Food Chem.* **1992**, *40*, 2260−2262.
(23) Gomes, T.; Caponio, F. Evaluation of the state of oxidation of olive-pomace oils. Influence of the refining process. *J. Agric. Food Chem.* **1998**, *46*, 1137−1142.
(24) Gomes, T.; Caponio, F. A study of oxidation and polymerization compounds during vegetable oil refining. *Riv. Ital. Sostanze Grasse* **1996**, *73*, 97−100.
(25) Hoffman, G. Refining. In *The Chemistry and Technology of Edible Oils and Fats and their High Fat Products*; Taylor, S. L., Ed.; Accademic Press: London, U.K., 1989; pp 139−200.

Received for review November 18, 2002. Revised manuscript received April 16, 2003. Accepted April 22, 2003.

JF026136T