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Changes in composition and quality of sunflower oils during extraction and degumming

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RESUMEN

Cambios en composición y calidad de aceites de girasol durante su extracción y desgomado.

Se estudió la influencia de las distintas etapas del procesamiento sobre la composición y calidad de aceite crudo de girasol a través de la determinación de la composición de ácidos grasos, contenido de fósforo y metales, índice de acidez, índice de peróxido, valor de p-anisidina, tocoferoles, compuestos polares, ceras y estabilidad oxidativa. No se apreció durante el procesamiento ningún cambio significativo en la composición de ácidos grasos y tocoferoles. Los contenidos de algunos compuestos minoritarios como metales, ceras y fosfolípidos resultaron apreciablemente afectados con los procesos de extracción y desgomado con agua. Los aceites de girasol obtenidos por extracción con hexano presentaron un grado de deterioro inicial superior y una mayor estabilidad oxidativa con respecto a los aceites obtenidos por prensado.

PALABRAS-CLAVE: Aceite de girasol - Calidad - Desgomado - Estabilidad oxidativa - Extracción.

SUMMARY

Changes in composition and quality of sunflower oils during extraction and degumming.

The influence of different stages of processing on composition and quality of crude sunflower oil was studied through the determination of fatty acids, metal and phosphorus contents, free fatty acids, peroxide and anisidine values, tocopherols, polar compounds, waxes and oxidative stability. No significant changes in the content of individual fatty acids and tocopherols were observed during processing. The composition of some minor constituents as metals, waxes and phospholipids was strongly affected by the extraction and water degumming processes. Sunflower oils obtained by hexane extraction presented a higher initial deterioration but also a higher oxidative stability than oils obtained by pressing.

KEY-WORDS: Degumming - Extraction process - Oxidative stability - Quality - Sunflower oil.

1. INTRODUCTION

Crude sunflower oil is obtained from partially dehulled seeds by mechanical pressing followed by hexane extraction and water degumming (Figure 1). Quality and stability are the major factors in the production, acceptance and marketing of vegetable oil products. These properties depend mainly on seed quality, seed treatment prior to extraction, extraction method and processing conditions. They are influenced by the presence of some minor components, as free fatty acids, tocopherols, phospholipids, trace metals and waxes, which have pro or antioxidant properties. Processing of oils cause alterations in their chemical composition, affecting their quality and oxidative stability.

Recently, several authors have investigated the influence of industrial processing, especially the refining process, on quality and stability of different vegetable oils as corn, soybean, olive, rapeseed, and sesame oil (Hopia, 1993; Pérez-Camino *et al.*, 1993; Kamal-Eldin and Appelqvist, 1995; Rade *et al.*, 1995; Smouse, 1995; Ferrari *et al.*, 1996, 1997; Ruiz-Méndez *et al.*, 1997; Abou-Gharbia *et al.*, 1997; Gomes and Caponio, 1997,1998). However, there are relatively few studies in the literature concerning the evolution of sunflower oil constituents during processing. In particular, little is known about the changes in oil quality caused by extraction and degumming. Composition of sunflower oils has been reviewed by Gunstone *et al.* (1994). Effect of pressing conditions on nonrefined sunflower oil has been presented by Turkulov *et al.* (1998) and the influence of water degumming in phosphatide content has been discussed in a previous paper (Crapiste *et al.*, 1998). On the other hand, some changes during refining have been reported by Karaali (1985), Mariani and Fedeli (1988), Hopia (1993), Dimic *et al.* (1994), Ruiz-Méndez *et al.* (1997), and Recseg *et al.* (1998).

The aim of this work was to investigate the effect of the extraction method, pressing or solvent extraction, and the water degumming process on composition, quality and oxidative stability of crude sunflower oil.

2. EXPERIMENTAL PROCEDURES

2.1. Sunflower oil

Samples (S1-S6) of sunflower oil, from different seed lots and at different stages of the process were

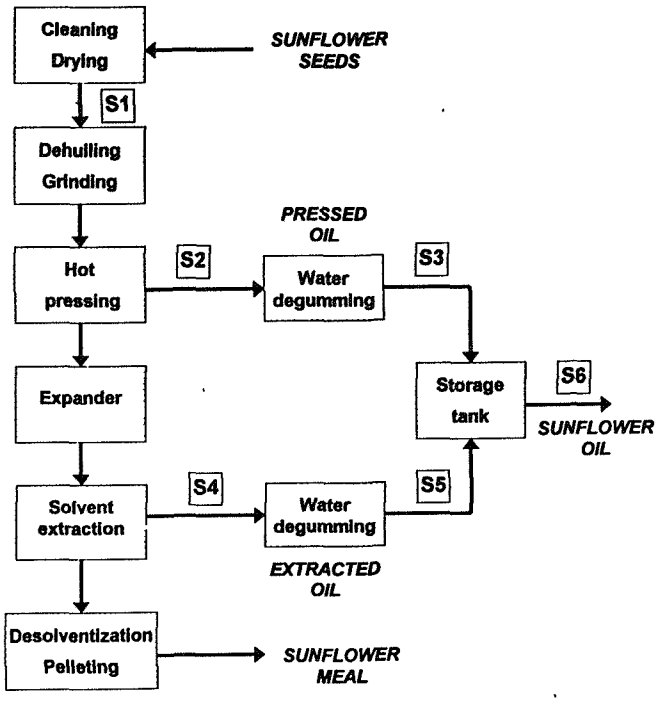

Figure 1
Scheme of the production of crude sunflower oil.

obtained from an industrial plant (Figure 1). Cleaned and partially dehulled sunflower seed, at approximately 8% moisture and 10% residual hull content, was used as raw material. Two independent sets of sunflower oils obtained by pressing or solvent extraction, with and without water degumming were analyzed. A sample from the storage tank was also analyzed for one set. In addition, cold pressed oils obtained in the laboratory from each seed lot were used for comparison.

2.2. Methods

Several analytical methods were used to determine oil composition, quality and stability. Acidity or free fatty acids, peroxide value, p-anisidine value and total phosphorus content were determined by standard AOCS official methods (AOCS, 1993). Trace metals (iron and copper) were measured by flame atomic absorption with a GBC 902 Atomic Absorption Spectrometer (GBC Scientific Equipment, Victoria, Australia). The oxidative stability index (OSI), represented as induction time in hours, was measured with a Metrohm 679 Rancimat (Metrohm, Switzerland) at 98°C and 20 L/h airflow.

Fatty acids. Fatty acid composition was determined by gas chromatographic analysis according to IUPAC 2.301-2.302 standard methods (IUPAC, 1992). The fatty acids methyl esters were separated on a 10% GP-DEGS-PS (L = 2 m, di = 0.32 cm) tubular column and quantified by flame ionization detection (FID) using a Varian 3700 gas chromatograph (Varian Associates Inc., Palo Alto, CA).

Tocopherols. Tocopherol content was measured by high-performance liquid chromatography using AOCS method Ce 8-89 (AOCS, 1993). A Varian Vista 5500 HPLC system (Varian Associates Inc., Palo Alto, CA) with a fluorescence detector set at 290-330 nm and a LiChrosorb Si-60 (250 x 4 mm 5 μm particle size) column (Merck, Darmstadt, Germany) was used.

Phospholipids. Quantitative determination of phospholipids was carried out by enrichment using diol solid phase extraction cartridges (J.T. Baker Inc., Phillipsburg, NJ) and subsequent analysis by high-performance liquid chromatography (Carelli *et al.* 1997). A Varian Vista 5500 HPLC system (Varian Associates Inc., Palo Alto, CA) with an ultraviolet detector set at 206 nm and a Micropak Si-10 (300 x 4 mm, 10 μm particle size) column (Varian Associates Inc., Palo Alto, CA) or a LiChrosorb Si-60 (250 x 4 mm, 5 μm particle size) column (Merck, Darmstadt, Germany) was used.

Waxes. Wax composition was determined by separation with silica gel chromatographic column and analysis by gas chromatography. A Varian 3700 GLC with FID detector and on-column injection (Varian Associates Inc., Palo Alto, CA), a HP5, 11 m x 0.32 mm (0.52 μm) capillary column (Hewlett Packard, Palo Alto, CA) and a Millenium 2010 data processor (Millipore Corporation, Milford, MA) were used.

Polar compounds. Separation of polar compounds was carried out by column chromatography with silica gel and verified by thin layer chromatography according to AOCS method Cd 20-91 (AOCS, 1993). Polar compounds were analyzed by high-performance size-exclusion chromatography (Dobarganes *et al.,* 1988). A Waters HPLC, two 500 and 100 A° Ultrastyragel (0.77 x 30 cm) columns connected in series, a refractive index detector and a Millenium 2010 Chromatography Manager (Millipore Corporation, Milford, MA) were used.

3. RESULTS AND DISCUSSION

Each separate sample was analyzed in duplicate or triplicate, and the results are summarized in Tables I and II. The main fatty acids in sunflower oils are palmitic, stearic, oleic, and linoleic (Table III). No appreciable effects of extraction method and degumming on fatty acid composition were observed.

Autoxidation and hydrolysis of lipids are the major causes of oil deterioration. The quality of crude oils varied with processing, as measured by both standard analysis (peroxide value, acidity, and

anisidine value) and polar compound analysis. Acidity or free fatty acids (as % oleic acid) as well as peroxide value (PV) and p-anisidine value (AV) increased gradually throughout the extraction process. Oils obtained by hexane extraction presented a higher initial acidity than oils obtained by pressing. Karaali (1985) reported an opposite effect, with crude pressed oil containing more free fatty acids than extracted oils, and concluded that pressing conditions encourage hydrolysis. Acidity is reduced by degumming, particularly in extracted oils. PV and AV also varied, being lower in pressed oils than in extracted oils, owing to the generation and decomposition of hydroperoxides during solvent extraction. In general, PV increased while AV remained approximately constant or increased slightly with degumming, suggesting that some decomposition of hydroperoxides without removal of secondary decomposition products occurred during this stage. These results indicate that oxidation increased progressively during solvent extraction and degumming. On the other hand, oils obtained by cold-pressing showed the best initial quality, with relatively low hydrolytic and oxidative alterations.

Table I

General characteristics of crude sunflower oils (set 1)

Analytical determination	Pressed Lab - Cold	Pressed		Hexane extracted		Tank
		—	Degummed	—	Degummed	Degummed
Acidity (% oleic acid)	1.13	1.27	1.21	1.68	1.35	1.25
Peroxide value (meq/kg)	0.83	0.85	4.06	3.05	5.01	5.49
Anisidine value	0.39	1.50	1.89	2.07	2.19	1.93
Polar compounds (g/kg)	86.0[a]	75.2[a]	60.7[a]	98.3[a]	73.1[a]	75.3[a]
	38.8[b]	43.4[b]	37.1[b]	45.3[b]	44.0[b]	39.7[b]
Metal content (ppm)						
Cu	tr.	1.8	tr.	1.5	tr.	tr.
Fe	8.0	5.4	3.1	7.7	3.3	3.9
Total phosphorus (mg/kg)	33	932	83	662	140	85
Phospholipids (g/kg)	tr.	7.37	1.32	11.95	2.07	1.27
Tocopherols (mg/kg)						
α-tocopherol	722	669	692	688	739	700
β-tocopherol	30	27	27	33	29	27
OSI (at 98°C)	10.2	20.2	12.5	29.9	16.3	10.5

Values are means of two-three replicate determinations.
[a] calculated by difference from the proportion of the non-polar fraction, [b] weight recovered by solvent elution.

Table II

General characteristics of crude sunflower oils (set 2).

Analytical determination	Pressed Lab - Cold	Pressed		Hexane extracted	
		—	Degummed	—	Degummed
Acidity (% oleic acid)	0.94	0.68	0.62	0.98	0.75
Peroxide value (meq/kg)	1.13	9.51	12.66	11.72	3.66
Anisidine value	0.39	0.97	1.03	1.46	1.35
Polar compounds (g/kg)	44.9[a]	83.9[a]	52.1[a]	65.2[a]	43.8[a]
	30.0[b]	40.5[b]	30.6[b]	38.9[b]	25.0[b]
Metal content (ppm)					
Cu	tr.	1.6	tr.	1.5	tr.
Fe	9.2	10.8	3.6	15.1	4.4
Total phosphorus (ppm)	27	523	77	441	44
Phospholipids (g/kg)	tr.	3.91	1.01	7.90	1.37
Total waxes (mg/kg)	205	409	405	464	433
Tocopherols (mg/kg)					
α-tocopherol	611	585	597	634	647
β-tocopherol	34	34	35	37	39
OSI (at 98°C)	8.0	20.1	12.4	22.4	12.9

Values are means of two-three replicate determinations.
[a] calculated by difference from the proportion of the non-polar fraction, [b] weight recovered by solvent elution.

Table III
Fatty acid composition of sunflower oils (%).

Fatty acid	Set 1 (*)	Set 2 (*)
16:0	6.2 - 6.3	6.3 - 6.4
18:0	3.1 - 3.4	3.1 - 3.5
18:1	21.8 - 22.4	23.0 - 23.4
18:2	68.2 - 68.3	66.9 - 67.1

* Limit values for five samples with different processing (by duplicate)

Determination of polar compounds by means of HPSEC provided further information. The method determines those specific polar compounds associated with hydrolytic and thermooxidative alterations in edible oils: polymers and dimers of triglycerides, oxidized triglycerides, diglycerides, and free fatty acids. It provides appropriate measurement of the state of degradation of the oil and has been successfully used to evaluate deterioration during processing and autooxidation (Hopia, 1993; Pérez-Camino *et al.,* 1993; Ruiz-Méndez *et al.,* 1997; Gomes and Caponio, 1997, 1998). It can be seen (Tables I and II) that fractions of polar compounds recovered by solvent elution were lower than those calculated by difference from the proportion of the non-polar fraction, according to AOCS method for refined oils. This can be attributed to the presence of meal residues, some non-lipid compounds, phospholipids, pigments and other minor components that are strongly retained in the chromatographic column. Since these components of crude oils do not constitute the polar fraction under study, it appears that values obtained by measurements of total polar compounds are more representative than those evaluated from the weight of the non-polar fraction. Polar compound levels changed with the process, being significantly lower in cold pressed oils and decreasing with degumming (Tables I and II). As it is shown in Figure 2, the polar fraction consisted mainly of oxidized triglycerides (OTG), diglycerides (DG), and free fatty acids (FFA). No appreciable amounts of dimers and polymers of triglycerides were detected in the oils, an expected result since polymerization due to thermal degradation occurs at high temperatures. The most significant change is observed in the concentration of OTG, associated with oxidative alteration, being higher in pressed than in extracted oils and lower in degummed oils. This can be explained by the fact that pressing conditions enhance oxidation due to more contact with air. In addition, some settling of OTG with phospholipids and other materials may occur during degumming. DG and FFA levels, related to hydrolytic alteration, changed with the extraction process following the same pattern than acidity and decreased slightly in degummed oils. This indicates

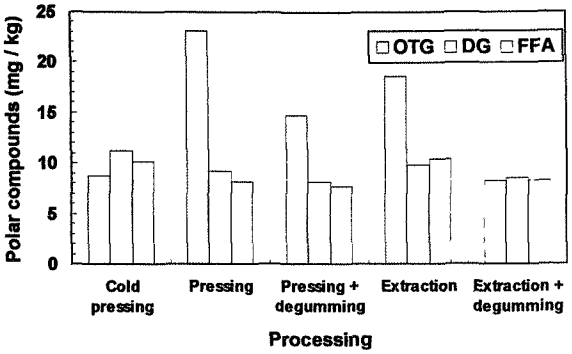

Figure 2
Effect of processing on composition of polar compounds in sunflower oils. OTG = oxidized triglycerides, DG = diglycerides, FFA = free fatty acids.

that no significant hydrolytic alteration occurs during water degumming.

Trace metals, particularly copper ions, are known to be effective prooxidants in lipid oxidation, so they are undesirable components from the point of view of oxidative stability. Metal content in crude sunflower oils was extremely high, reducing noticeably with degumming. Solvent extracted oils showed similar concentrations of copper and higher concentrations of iron than pressed oils. The iron content in crude oil is due to natural iron present in oilseeds (mainly bounded to proteins, phospholipids and other minor constituents) and possible contamination from metallic process equipment, as iron soap of fatty acids, which has prooxidant activity (List *et al.,* 1978; Karaali, 1985). Water degumming reduced significantly the iron concentration, while the copper content decreased to the limit of detection in degummed oils. This may indicate that, apart from the cold-pressed oil, the higher percentage of ions was complexed with phospholipids.

Phospholipids are natural components of oilseeds that pass to oil during extraction. They are partially removed at the crushing plant by degumming with water and almost completely removed from crude oils during refining. The major phospholipids in sunflower oils are phosphatidylcholine (PC), phosphatidylethanolamine (PE), phosphatidylinositol (PI) and phosphatidic acid (PA). As it is shown in Figure 3, crude oils from seed lot 2 showed lower phospholipid contents than oils from seed lot 1. The phospholipid content was higher and enriched in PE and PC for solvent extracted oils, while cold pressed oils contained very small amounts of phosphatides. It has been reported elsewhere that crude pressed oil contains less phospholipids than extracted oils (Gunstone *et al.,* 1994; Dimic *et al.,* 1994; Crapiste *et al.,* 1998). In addition, Turkulov *et al.* (1998) observed that a more intensive hydrothermal treatment of the seed

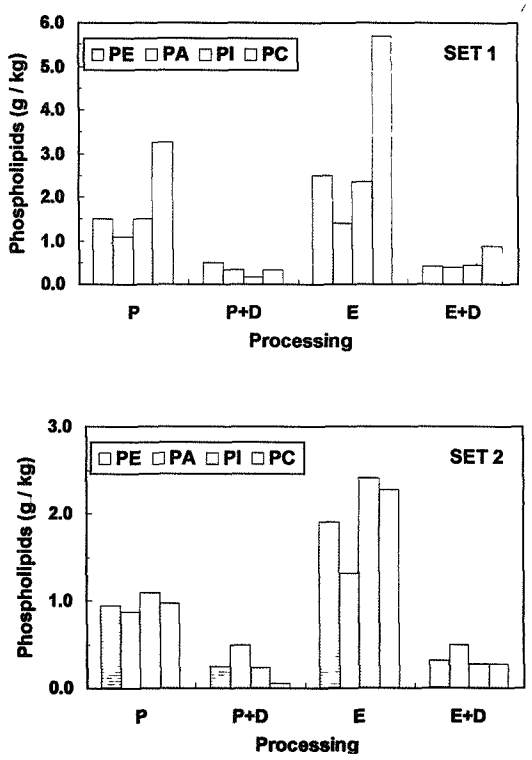

Figure 3
Effect of processing (P - pressing, E - extraction, D - degumming) on phospholipid content in sunflower oils. PC = phosphatidylcholine, PA = phosphatidic acid, PE= phosphatidylethanolamine, PI = phosphatidylinositol.

produced a higher phospatide content in the oil. It can be concluded that the phospholipid content increases with thermal treatment and intensity of extraction. Most of sunflower oil phospholipids are hydratable and can be removed from the oil by degumming with water, the efficiency of the process depending on phosphatide composition and treatment conditions (Crapiste *et al.,* 1998). It can be observed that degummed oils contained significantly higher percentages of PA and lower percentages of other phospholipids, particularly PC, than original crude oils (Figure 3). Previous studies indicate that PC is almost fully hydratable while PA is nonhydratable when complexed with calcium or magnesium, and PC and PI hydrate considerably faster than PE and PA (Crapiste et al, 1998).

Crude sunflower oils have very high wax contents, up to 1500 ppm depending on the seed and the extraction technology. Waxes are mainly derivatives of long chain fatty alcohols with long chain fatty acids, ranging from 36 to 50 carbon atoms or even more (Mariani and Fedeli, 1989; Recseg *et al.,* 1998). In our case the wax content of the oil was relatively low, the higher percentages corresponding to the fraction C40-C41 followed by C46, C44 and C48 (Table IV). Although the wax fraction consisted mainly of even-carbon numbered wax esters, some concentration of odd-numbered waxes, particularly C39 and C41, was also determined. Wax content and composition were affected by the extraction method while degumming has a little or no effect on the total wax content. Solvent extraction yielded oils with a higher concentration of waxes, especially those components with carbon number higher than 42. Most short-chain waxes are soluble in oils, but long-chain compounds may cause turbidity during oil storage and must be eliminated by winterization in the refining process. The wax content decreased significantly in laboratory cold-pressed oils, particularly in the fractions of high molecular weight, indicating that wax extractability depends strongly on extraction temperature.

Table IV
Wax composition of sunflower oil (%).

Wax	Pressed Lab - Cold	Pressed		Hexane extracted	
		—	Degummed	—	Degummed
C36	13.6	7.6	7.4	7.8	7.2
C37	7.3	4.9	4.7	5.4	4.4
C38	2.0	0.9	1.0	1.7	1.2
C39	1.3	0.7	0.8	1.0	0.9
C40	26.8	12.5	12.3	12.5	11.3
C41	30.2	18.8	15.6	17.5	14.1
C42	5.4	6.6	5.2	5.4	5.3
C43	2.0	1.4	1.4	1.9	1.5
C44	3.5	20.3	20.0	13.8	16.2
C45	0.6	0.6	0.5	0.9	0.8
C46	3.4	17.1	22.0	18.5	21.9
C48	4.0	8.6	9.1	13.8	15.2
Wax content (mg/kg)	**205**	**409**	**405**	**464**	**433**

Values are means of two independent determinations

Sunflower oils contained about 650-750 mg/kg of tocopherols, natural antioxidants present in the oil, mainly as α-tocopherol (>95%) and β-tocopherol. Tocopherol contents were slightly higher in oils obtained by cold pressing in the laboratory than in pressed oils, suggesting that only a small amount of the initial tocopherol is altered during the industrial extraction process. Certain differences were observed since extracted oils also showed a slightly higher concentration of tocopherols than industrially pressed oils. However, it should be taken into account that pressed oils contain more non-lipid fraction from the seed. Therefore, it may be concluded that the effect of processing on tocopherols was practically negligible.

Accelerated oxidation tests using the Rancimat method are extensively used to evaluate the oxidative stability of fats and oils. The oxidative stability of vegetable oils was shown to be dependent on extraction method and processing (Smouse, 1995; Kamal-Eldin and Appelqvist, 1995; Abou-Gharbia *et al.,* 1997). In this study solvent extraction yielded more-stable oils than pressing, and degumming significantly reduced the oil stability (Tables I and II). Pressed and extracted oils had approximately the same concentration of unsaturated fatty acids, natural antioxidant (tocopherols) and some prooxidants (metals and free fatty acids) while degumming reduced the amount of prooxidants. Therefore, the differences in oxidative stability can be attributted to the concentration of phospholipids, which decreased in the same order: extracted, pressed, degummed, and cold-pressed oils. Phospholipids have strong antioxidant effects due to their synergistic action with tocopherols, their metal scavenging activity, and also to their catalytic activity to decompose hydroperoxides (Smouse, 1995; Carelli *et al.,* 1997).

As expected, composition of crude mixed oil from the storage tank presented intermediate values in most analysis. They were not equal to the means of pressed and extracted oils because they are mixed in different proportions. On the other hand, settling of non-lipid materials and some minor components (as phospholipids, waxes, and polar compounds), along with some additional deterioration, may occur during storage.

4. CONCLUSIONS

The composition of minor constituents, quality and oxidative stability of crude sunflower oils were strongly affected by the extraction process. Phospholipids, waxes and probably some trace metals are better extracted by solvent than by pressing and have a higher extractability under hot conditions. Higher concentrations of less-hydratable phospholipids and long-chain winterizable waxes were found in solvent extracted oils. Content and distribution of individual fatty acids and tocopherols were not significantly affected by processing, aside from the fact that extracted oils had a slightly higher concentration of tocopherols. Extracted oils, which showed a higher initial deterioration, also presented a higher oxidative stability mainly due to the antioxidant effect of phospholipids. The degumming process produced a decrease in acidity, polar components, and trace metals and an increase in peroxide and anisidine values, yielding considerably less-stable oils. Oils obtained by cold-pressing showed the best quality, with a small oxidative alteration, only traces of phospholipids and lower wax contents. However, they were less stable due to the low phospholipid content.

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