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Effects of a membrane-based soft purification process on olive oil quality

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Abstract

Because of the high temperatures applied in classical refining processes, many quantitative and qualitative changes occur in the chemical compositions of oils. This causes refined olive oil to lose many of its organoleptic and nutritional properties. Research has been carried out to ascertain the influence of a soft purification process, based on oil conditioning, to form micronic aggregates which were subsequently removed by a microfiltration operation. The quasi total acidity and the resulting soaps were removed in a single microfiltration step. The deacidified and filtered oils showed similar peroxide values and the variation of the specific extinction around 270 nm (ΔK) was slightly improved. The process lowered the monoglyceride contents up to 78% while the diglyceride contents were just slightly decreased. The various sterol components were also lowered (in the range 36–50%) upon treatment. Phenolics were the most affected components; reductions reached 93%. The partitioning of such components between the bulk triglyceridic medium and the aggregate phase are considered with regard to their properties. Despite the low operating temperature, the sensory evaluation of the deacidified and filtered oils still showed poor organoleptic characteristics.

Keywords: Olive oil; Deacidification; Microfiltration; Minor components; Quality

1. Introduction

Among animal and vegetable fats, olive oils are particularly recommended for human nutrition because of their health benefits. According to international regulations, many quality categories are established. The less valuable virgin oil, lampante virgin olive oil, which shows high acidity levels (>3.3%) is not recommended for human consumption and must be refined. Unfortunately, due to bad practices, especially in south Mediterranean countries, a great part of the olive oil production suffers from poor quality and requires further purification to meet international standards.

During refining operations quantitative and qualitative changes take place, especially in components responsible for quality. High temperatures seem to be the most harmful parameter and are responsible for hydrolytic, oxidative and polymerisation alterations (Pérez-camino, Ruiz-mendez, Marquez-ruiz, & Dobarganes, 1993; Ruiz-Mendez, Marquez–Ruiz, & Dobarganes, 1997). Structural changes are also related to the heating especially during bleaching (Schulte, 1995) and deodorisation (Cert, Lanzon, Carelli, Albi, & Amelotti, 1994). Devinant, Scamaroni, and Naudet (1980) and Jawad, Kocchar, and Hudson (1983) reported that, during physical refining, the stereoisomerisation and polymerisation depend on time and the temperature. Some

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sterols can undergo dehydration during bleaching (Schulte, 1995) and deodorization (Cert et al., 1994), so β sitosterol can form 3,5 stigmadiene, which is usually used as a marker of refined olive oils. Total sterols are also reported to be lower after neutralisation (Kochhar, 1983). Pasqualone and Catalano (2000) reported that neutralized oils showed losses of up to 50% in total sterols. Gomes (1992) and Catalano, De Leonardis, and Comes (1994) reported increases of diglycerides in refined olive oil.

Most of these alterations are temperature-dependent. In previous works we have developed a soft membrane-based lampante olive oil deacidification process which operates at low temperatures (20–25 °C) (Hafidi, Pioch, & Ajana, 2004a, 2004b). This was expected to preserve the sensitive and bioactive components in the olive oils. In the present study, the impact of such a process on some minor components and on the organoleptic characteristics of the purified oils was ascertained.

2. Materials and methods

2.1. Oil conditioning

Lampante virgin olive oil (LVOO) samples were neutralized at room temperature with a sodium hydroxide aqueous solution and then microfiltered as described in previous works (Hafidi et al., 2004a, Hafidi, Pioch, & Ajana, 2004b).

2.2. Analytical methods

In order to check the chemical composition of both starting and refined oils, French standard procedures (AFNOR, 1984) were applied: Phosphorus NF T 60–227; Soaps NF T 60–217; Free fatty acids NF T 60–204; Water NF T 60–367; Peroxide value NF T 60–220; UV absorbances NF T 60–223.

The compositions of fatty acids and sterols were determined according to the methods recommended by EC regulations (EU regulation No. 2568/91, 1991). After transmethylation, the fatty acids were analyzed using a capillary column SP-2330, 30 m × 0.32 mm × 0.2 μm, with helium as carrier gas, flow-rate 200 cm^3/s under isothermal conditions, at 185 °C. Detector (FID) and injector (Split) temperatures were 220 °C.

After extraction and fractionation of the insaponifiable fraction of the oil by thin-layer chromatography, the sterols were silylated and analyzed using SE-54 (Supelco) 30 m, 0.25 mm internal diameter capillary column under the following conditions: the oven initial temperature (180 °C) was maintained for 8 min before beginning a rise at 5 °C/min to 260 °C which was maintained for 15 min; injector and detector temperatures were 280 and 290 °C, respectively.

Partial glycerides were analyzed by gas chromatography according to the method described by Plank and Lorbeer (1992). A DB1 WTHN capillary column was used (7 m length, 0.32 mm internal diameter). The carrier gas (helium) flow rate was set at 2.9 ml/min; the temperature was held at 100 °C for 3 min before a rise at 10 °C/min, to 340 °C which was maintained for 5 min. Hexatriacontane was used as an internal standard. The silylation was performed with the bis (trimethylsilyl) trifluoroacetamide (BSTFA) and trimethylchlosilane (TMCS).

Analysis of polyphenols consisted of two steps: (i) the compounds were extracted with methanol/water mixtures as described by Montedoro, Servili, Baldioli, and Minati (1992), and then (ii) a spectrophotometric assay was used to estimate their relative amounts using the Folin–Ciocalteu reagent. The content of phenolics was expressed as ppm of caffeic acid equivalents.

Sensory analysis was carried out by an analytical panel of the Cereol company, Spain; according to European Official Methods EC/2568/91 (EU regulation No. 2568/91, 1991).

3. Results and discussion

3.1. Effect on the physicochemical characteristics

Results in Table 1 showed the almost complete deacidification of the LVOOs after conditioning and crossflow filtration. 20% NaOH allowed better neutralization. In all cases the filtered oils were soap-free; the phosphorus and water contents were also decreased. Deacidification and filtration did not improve the peroxide value which remained almost unchanged. The ΔK values were globally slightly improved. The sodium soaps resulting from the alkali treatment self-organize into close-packed multilamellar vesicles of submicronic sizes (Largueze, Pioch, & Gulik-Kryzwicki, 2002). Phospholipids are also reported to be eliminated in one operation with the soaps and are believed to play essential role in the aggregation mechanisms, especially in seed oils where their concentration are well above those observed in olive oils (Largueze et al., 2002; Pioch et al., 1996; Pioch, Largueze, Graille, Ajana, & Rouviere, 1998). Those aggregates are subsequently removed by a microfiltration operation. The total added water via the alkali solutions must be entrapped in the bilayer structures of the aggregates. The complete elimination of water from olive oil was recently found not to be very beneficial for oil oxidative stability (Ambrosone, Angelico, Cinelli, Di Lorenzo, & Ceglie, 2002; Lercker, Frega, Bocci, & Servidio, 1994). This, the elimination of natural antooxidants and the exposure to air during microfiltration may contribute to oxidation of the filtered oils and may explain the slight increase in the PV.

Table 1
Physicochemical characteristics of virgin and deacidified lampante olive oils

Olive oil samples		[NaOH]	[H_3PO_4]	FFA (%)	P (ppm)	Soaps (ppm)	Water content (%)	ΔK	PV (meq/kg)
LVOO1	Crude	–	–	3.67	15.8	–	0.16	0.026	26
	Deacidified	20%	–	0.12	5.2	nd	0.09	0.022	28
		20%	0.1%	0.08	2.4	nd	0.08	0.018	25
		40%	–	0.30	7.1	nd	0.08	0.024	27
		40%	0.1%	0.16	2.6	nd	0.08	0.022	33
LVOO2	Crude	–	–	5.12	10.5	–	0.20	0.040	35
	Deacidified	20%	–	0.10	3.4	nd	0.08	0.015	29
		20%	0.1%	0.06	2.2	nd	0.08	0.026	31
		40%	–	0.16	4.6	nd	0.06	0.031	37
		40%	0.1%	0.12	1.4	nd	0.06	0.042	32
LVOO3	Crude	–	–	7.20	12.7	–	0.13	0.067	52
	Deacidified	20%	–	0.12	2.9	nd	0.08	0.046	59
		20%	0.1%	0.08	2.3	nd	0.06	0.053	65
		40%	–	0.21	5.4	nd	0.06	0.064	57
		40%	0.1%	0.16	2.5	nd	0.08	0.059	66

Table 2
Influence of processing on the fatty acid composition of the LVOO 1

	Crude	NaOH 20%	0.1% H_3PO_4 + 20% NaOH	40% NaOH	0.1% H_3PO_4 + 40% NaOH
C 16 : 0	11.2	12.7	11.8	13.5	12.6
C 16 : 1	0.59	0.97	0.76	0.83	0.96
C 18 : 0	4.50	4.86	4.33	4.6	5.12
C 18 : 1	76.1	73.3	74.1	72.7	73.6
C 18 : 2	6.35	6.68	7.45	6.95	6.23
C 18 : 3	0.71	0.73	0.94	0.78	0.84
C 20 : 0	0.32	0.47	0.39	0.40	0.36
C 20 : 1	0.28	0.34	0.25	0.22	0.31

3.2. Effect on the total fatty acids and partial glycerides

As shown in Table 2, the deacidification process did not affect the total fatty acid composition. But, the partial glyceride, especially monoglycerides (MG), contents were lowered (Table 3). The MG content decreases between 44% and 78% depending upon the case. From the results, it is clear that 20% NaOH leads to important MG elimination. The lowest decrease in MG was observed when using 40% NaOH without a phosphoric acid pre-treatment. The diglycerides (DG) were just slightly decreased (2–18%). The use of 20% NaOH leads to more important elimination rates (9–18%) in comparison with the rates obtained with the 40% NaOH.

DGs result essentially from enzymatic hydrolysis of the triacylglycerols and, in a further step of the reaction, MGs are formed. According to the theory of the equimolar formation of FFA and DG, their molar contents in oil are expected to be identical, and the weight proportion of DG should be almost twice of that of FFA. Many researchers have noticed that this is not verified experimentally and this may due to the differences in partitioning of FFA and DG between oil and the residue and also to the solvent and mode of the oil extraction (Brül, 1997). As far as our process succeeds in completely removing the FFA while the DG undergoes only a slight decrease, the ratio FFA/DG can be used to differentiate the virgin olive oils from the refined ones. The relatively high elimination of the MGs subsequent to the neutralization, in comparison to the DGs is most likely related to their surface activity. In fact, MGs have higher surface activity than DGs and are commonly used as emulsifiers in the food industries. Gaonkar and Borwankar (1991) reported a competitive adsorption behaviour at the vegetable oil–water interface between MG and lecithins and showed the possibility of forming mixed micelles .

3.3. Effect on the sterol composition

The identification and quantitation of minor components, naturally present in edible oils or generated as a consequence of refining, are utilized for characterization purposes as well for the detection of frauds. The unsaponifiable fraction of olive oil contains a wide variety of minor constituents which can be subdivided into tocopherols, phenols, flavour compounds, hydrocarbons and sterols. Some of these substances, especially tocopherols and phenolic compounds, are reported to exert beneficial effects on health (Wahrburg, Kratz, & Cullen,

Table 3
Influence of processing on partial glyceride contents

Olive oil samples		[NaOH]	[H_3PO_4]	Monoglycerides (%)	Diglycerides (%)
LVOO1	Crude	–	–	0.75	4.56
	Deacidified	20%	–	0.28	4.15
		20%	0.1%	0.16	3.85
		40%	–	0.32	4.28
		40%	0.1%	0.26	4.23
LVOO2	Crude	–	–	0.93	3.53
	Deacidified	20%	–	0.34	3.14
		20%	0.1%	0.28	3.05
		40%	–	0.52	3.45
		40%	0.1%	0.31	2.86
LVOO3	Crude	–	–	1.49	4.75
	Deacidified	20%	–	0.45	4.13
		20%	0.1%	0.37	3.87
		40%	–	0.73	4.18
		40%	0.1%	0.56	4.28

2002). Others improve the stability of the oil, and some are responsible for its unique taste and flavour (Kiristakis, 1998; Servili & Montedoro, 2002; Wahrburg et al., 2002).

Sterols constitute the major portion of the unsaponifiable matter of most vegetable oils. They exist mainly as free forms or may be esterified with fatty acids. The sterol composition constitutes the analytical method of choice to determine the plant species from which the oil has been extracted. EC regulations assign limits for total sterols to the different categories of olive oils and limits for their qualitative and quantitative composition to protect their quality and authenticity (EU regulation No. 2568/91, 1991).

The impact of the process which comprises a cold deacidification step, on the sterol composition is illustrated in Table 4. The separation of the vesicle-like and soap-made macrostructures formed in the oil by microfiltration resulted in a reduction of the total sterol contents. The elimination rates range between 36% and 50%. No clear differences were observed between the treatments whereas the reduction rates seem to increase slightly with the oil initial acidity (36–39% for the LVOO1, 37–45% for the LVOO2 and 41–50% in the case of LVOO3). The reductions applied to all the different sterol compounds, with relatively close proportions. The lowest reductions occured with stigmasterol. These findings are in complete agreement with results reported by many authors (De Blas & del Valle Gonzalez, 1996; Grob, Lanfranchini, & Mariani, 1990; Kochhar, 1983; Pasqualone & Catalano, 2000). In fact, conventional refining is known to affect the sterol content; all the different steps (degumming, neutralization, bleaching, hydrogenation, deodorization and steam refining) are involved. The reported reductions caused by the conventional alkali treatment operation range from 30% to

Table 4
Influence of processing on the sterol composition

Olive oil samples		[NaOH]	[H_3PO_4]	Total sterols (ppm)	Campestérol (ppm)	Stigmasterol (ppm)	Δ7Campestérol (ppm)	β Sitosterol (ppm)	Δ5Avenastérol (ppm)
LVOO1	Crude	–	–	2610	84.3	32.6	84.4	2139	270
	Deacidified	20%	–	1604	44.4	26.5	62.3	1288	165
		20%	0.1%	1559	44.6	22.7	49.0	1365	185
		40%	–	1557	53.0	24.3	64.5	1331	156
		40%	0.1%	1644	41.7	28.1	74.5	1283	158
LVOO2	Crude	–	–	2216	68.9	30.2	76.3	1875	232
	Deacidified	20%	–	1365	46.1	25.7	55.8	1137	163
		20%	0.1%	1143	38.9	23.0	46.3	1048	176
		40%	–	1245	35.6	21.6	42.9	990	149
		40%	0.1%	1295	42.3	26.2	44.8	1005	133
LVOO3	Crude	–	–	2766	97.3	44.1	102.6	2473	277
	Deacidified	20%	–	1749	44.9	36.4	69.4	1346	143
		20%	0.1%	1572	52.7	33.2	57.0	1206	136
		40%	–	1688	68.4	37.6	66.2	1420	149
		40%	0.1%	1634	62.2	30.3	60.5	1311	129

50% (De Blas & del Valle Gonzalez, 1996; Grob et al., 1990; Pasqualone & Catalano, 2000). EC regulations stipulate that, for virgin olive oils, the stigmasterol content must not exceed the campesterol content. Gutierrez et al. reported, for the first time, a high negative correlation between the stigmasterol content and the sensory properties (Guttierrez, Varona, & Albi, 2000); oils with more stigmasterol than campesterol are organoleptically graded as lampante. In fact, refining reverses these proportions but, in our case, although the stigmasterol elimination rates were the lowest, its contents remained lower than those of the campesterol.

3.4. Effect on the polyphenol contents

The total polyphenol contents were greatly reduced in all the oil samples after deacidification and tangential flow filtration (Table 5). In most cases, the reductions exceeded 80%. 20% NaOH seems to cause more important elimination than 40% NaOH. Visibly, after the neutralisation, phenolic components markedly partitioned toward the water phase entrapped in the sodium soap aggregates. In fact, phenolics are polar compounds and many of them are weak acids, so they can easily be removed from the oils with aqueous solutions, especially when neutralized. The reduction of phenolic contents is detrimental to oil oxidative stability and organoleptic characteristics. Many authors report that the concentration of phenolic compounds, evaluated colorimetrically and expressed as total phenols, was highly correlated with the shelf life of virgin olive oil, as estimated using accelerated methods such as AOM and rancimat (Chimi, Cillard, Cillard, & Rahmani, 1991; Ninfali, Aluigi, Bacchiocca, & Magnani, 2001; Papadopoulos & Boskou, 1991). Other researchers have indicated that some sensory properties are connected with the total phenol concentration (Gutiérrez Rosales, Perdiguero, Gutiérrez, & Olias, 1992).

The elimination of different minor components from vegetable oils, subsequent to the neutralisation, has been extensively studied; however, the mechanisms of such partitioning have not been investigated. Soap molecules, resulting of the alkali treatment, are amphiphilic, so they may self-assemble, under specific conditions, into multilamellar close-packed vesicles formed with hundreds of bilayer stacks, entrapping water molecules between the polar heads of the soap molecules (Pioch et al., 1998). We believe that the elimination of a portion of the MG, sterols and polyphenols is facilitated either by their polar character or, mainly, by an enhanced solubilization by the soap aggregates. In fact none of these components is known to be eliminated to such an extent by simple water washing.

The effects of conventional refining operations on vegetable oil quality have been widely studied. In the case of virgin olive oils, only the lampante category has to undergo a refining process to improve its acidity and/or its overall organoleptic characteristics. Completely refined olive oils are colourless, odourless and lose all the particular flavour which characterizes olive oils. To test the organoleptic quality of the lampante olive oils treated by the mild conditions of neutralization and microfiltration recommended in our process, three samples (crude oil and two corresponding deacidified samples) were graciously analyzed by the panel of the Cereol company, Spain (Table 6). Unfortunately, the overall sensory evaluation remained poor (score: ~2); our process did not succeed in improving the flavour of the deacidified oils. Almost all the other physicochemical characteristics showed the same behaviour as seen for the other oils.

Table 5
Polyphenol contents of lampante olive oils before and after deacidification. (μg caffeic acid equivalents/g oil)

Olive oils	Crude	Deacidified			
		20% NaOH	0.1% H_3PO_4 + NaOH 20%	40% NaOH	0.1% H_3PO_4 + NaOH 40%
LVOO1	84.5	15.0	12.7	21.0	16.4
LVOO2	77.5	11.2	13.4	17.3	15.3
LVOO3	178	18.0	12.5	22.5	14.8

Table 6
Sensory evaluation scores and some chemical characteristics of the LVOO4

	LVOO4	LVOO4/20% NaOH	LVOO4/40% NaOH
Sensory evaluation score	2.0	1.8	2.1
FFA (%)	4.0	0.15	0.21
PV (méqO_2/kg)	9.6	29.6	18.5
ΔK	0.016	0.016	0.016
Total sterols (ppm)	1963	1589	1528
Total aliphatic alcohols (ppm)	237	229	231

4. Conclusion

A partitioning of some olive oil minor components between the bulk triglyceridic media and the aggregate phases was found. Such behaviour involved all the studied components to different extents. The olive oil quality was affected as far as it was related to these components. The soft purification process allowed (at low temperature and in a single microfiltration operation) a complete deacidification of the lampante olive oils but did not succeed in improving the overall quality of the oils. Some desirable components, mainly phenolics, were eliminated during processing. Further investigations must focus on preventing or limiting the phenolic compound elimination and must to try to improve the organoleptic characteristics of the filtered oils. Fundamental studies on the physicochemical properties of the glyceridic media, the structure and the properties of the aggregates will be of a great interest for improving and controlling the partitioning of the different compounds and may help to set up an efficient bioactive-preserving process.

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