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Physical chemical and sensory characterization of olive oil of the region of Kairouan

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Abstract

This study focused on the characterization of olive oil produced in the Kairouan area (center of Tunisia) in order to install Protected Geographical Indication (PGI). Thus, chlorophylls, carotenoids, phenols and tocopherols contents, fatty acid composition, quality parameters and organoleptic profile were determined on 90 samples of extra virgin olive oil extracted from two different varieties of olives: Oueslati (cultivar grafted on oleaster varietie grown in Al Alaa province) and Chemlali (varietie grown in Essbika province). The results demonstrated that Oueslati olive oil samples were significantly ($p>0.05$) richer in antioxidant compounds such as tocopherols (252 ppm), carotenoids (1.73 ppm) and chloropylls (2.53 ppm) than olive oil harvest from chemlali varietie grown in the same geographical area. Moreover, fatty acid composition and sensory analysis of Oueslati olive oil samples were more interesting, balanced and specific. The higher oxidative stability characterizing Oueslati olive oil samples mainly due to their richness on antioxidant compounds and monounsaturated acids, and their specific taste, led to the installation of quality label (PGI) guaranteeing the authenticity and purity of this olive oil belonging to Al Alaa province

Keywords: Olive oil, Oueslati, Chemlali, geographical area, tocopherols, phenols, sensory analysis.

1. Introduction

Olive is a Mediterranean tree having a very ancient origin. His appearance and culture date back to prehistory. Olive oil was always appreciated for its quality. In contrast to other vegetable oils, it's consumed in its crude state without any refining process. Recently, virgin olive oil authenticity has become an important subject at the commercial and medical levels [1]. Nowadays, the tendency is the quality labels installation, such as Protected Geographical Indication (PGI), which can guarantee the authenticity and purity of the olive oil. This label is slightly less stringent than Protected Designation of Origin (PDO), but also demands that olive oil produced in the geographical region have the same name of this region. The geographical link must occur in at least one stage of production, processing or preparation. The quality labels installation encourage agricultural production, protect product names from misuse and imitation. It satisfy also consumers by giving them information concerning the specific characteristics of the products they are using on their daily nutrition. This authenticity is usually tested through the analysis of several parameters such as, fatty acids, triacylglycerol, sterol, volatile compounds and tocopherol contents. Moreover, chemical composition of virgin olive oil is influenced by genetic (variety), oil extraction technology and environmental factors such as climatological and edaphologic conditions. Olive oil traceability is based on the botanical factor and geographical origin identification [2]. Several researchers were

interested on olive oil minor components classified into two types: the unsaponifiable (non polar) fraction and the soluble (polar) one which includes the phenolic compounds. These elements are important from a biological perspective, despite their weak concentration in the oil [3]. Furthermore, previous studies revealed that monounsaturated fatty acids characterizing olive oil generate a reduction of cancer risks [4], [5]. Therefore, the current study was carried out to characterize the two varieties belonging to Kairouan area: Oueslati which is a cultivar grafted on Oleaster [6], [7], [8],[9] and Chemlali, in order to install a PGI. This region was chosen since it is one of the Tunisian regions mostly characterized by its important olive production and the olive area of Al Alaa province is a mountainous specific area.

2. Experimental

2.1Sampling

90 samples of extra virgin olive oil were obtained after cold extraction of two different varieties of olive fruits (*Olea europaea L.*) collected from three successive campaigns in the region of Kairouan: 45 samples belonging to Oueslati variety (cultivar grafted on oleaster varietie grown in Al Alaa province) and 45 samples belonging to Chemlali variety grown in Essbika province.

2.2 Methods

2.2.1Oxidative stability determination

To characterize olive oil of the Kairouan region, Free Acidity (FA), Peroxide Value (PV) and UV absorption (K_{270}, K_{232}) were determined according to the method of European Community Regulation [10].

2.2.2 Fatty Acid Methyl Esters Preparation and Analysis

Oil fatty acids were transformed after a transmethylation into their fatty acid methyl esters (FAMEs) according to the method described by COI [11]. The methyl esters were prepared by vigorous shaking of an olive oil solution in heptane (0.5 g in 4 mL) with 0.5 mL of 2 N methanolic KOH in a test tube with a screw cap. The mixture was centrifuged and the supernatant layer containing the methyl esters was used for gas chromatography (GC) analysis. FAMEs were analyzed by GC-FID chromatograph (model 6820 Agilent Technologies,Wilmington, DE, USA), equipped with a Carbowax (30 m × 0.32 mm × 0.32 µm) capillary column. The carrier gas was helium, with a flow of 1 mL/ min. Injector and detector temperatures were set at 230 and 250 °C, respectively. The injection volume was 5 µl.

2.2.3 Pigments contents determination

Chlorophylls and carotenoids contents were determined following the procedure of Minguez-Mosquera et al. [12]. In brief, 7.5 g of oil were weighed, dissolved in cyclohexane and taken to a final volume of 25 mL. Carotenoids and chlorophylls pigments were determined by measuring the absorbance at 470 and 670 nm, respectively. Pigment content was expressed using the following equations:

$$[\text{Chlorophylls (mg/kg)}]=Abs_{670}*10^6/613*1000*\text{density}$$
$$[\text{Carotenoids (mg/kg)}]=Abs_{470}*10^6/2000*1000*\text{density}$$

2.2.4 Tocopherols contents determination

Tocopherols contents were determined by high-performance liquid chromatography (HPLC) according to the standard method [13]. Oils were diluted in acetone and injected directly into C_{18} column. The mobile phase was constituted by 96 % Methanol/ acetonitrile (50/50) and 4 % water/ phosphoric acid (99:1) mixture. Tocopherols were detected at 294 nm by the UV-vis detector.

2.2.5 Phenols contents identification and quantification

Phenols identification and quantification was determinate by HPLC [14] equipped with a UV detector at 280 nm, with C18 reverse-phase column (4.6 mm x 25 cm) type ODS-2 5mm.

2.2.6 Sensory analysis

Sensory analysis was carried out by the official panel of National Office of Oil. The panel was approved by the International Olive Council. Panel test was used to distinguish olive oil from other edible vegetable oils evaluating positive and negative descriptors. According to the method described in COI regalements the odor or taste attributes (fruity, bitter and piquant intensities) were quantified using a dix-point intensity ordinal rating scale from 0 (no perception) to 10 (extreme). [15]

3. Results and discussion

3.1 Oxidative stability determination

Physicochemical parameters (FA, K_{270}, K_{232} and PV) of olive oil samples were analyzed to determine their oxidative stability. PV is a widely used measure of primary lipid oxidation indicating peroxides amount formed during oil oxidation. In addition, FA formation might be an important measure of rancidity of foods. In fact, FA resulting from the hydrolysis of triacylglycerides as well as the further decomposition of hydroperoxides, is one of the most important indicators of oil deterioration. Moreover, changes in UV absorption at 232 and 270 nm are associated with changes in conjugated dienes and trienes produced by the oxidation of polyunsaturated fatty acids. The higher the proportion of polyunsaturated fatty acids in the oil, the higher are the levels of conjugated dienes and trienes formed.

Table 1 showed low values for the regulated physicochemical parameters valuated (FA $\leq$ 0.8%, $K_{270} \leq 0.220$ and PV $\leq$ 20 meq. O_2. Kg^{-1}). All samples belonged to the ranges established for "extra virgin olive oil" (EVOO) category, as required by the European Community Regulation [10]. Samples extracted from Oueslati variety were characterized by a significantly ($P<0.05$) higher oxidative stability, since their FA, PV and UV absorption values were significantly lower than that observed in the samples from Chemlali one. Similar results were carried out by Ouini et al.[16]

Table 1: Oxidative stability parameters

	Olive oil samples	
Parameters	**Oueslati variety**	**Chemlali variety**
FA (% C18:1)	0.32b ± 0.04	0.34a ± 0.06
PV (meq.O_2.kg^{-1})	9.96b ± 0.78	10.66a ± 0.96
K_{232}	2.07b ± 0.09	2.13a ± 0.11
K_{270}	0.128b ± 0.01	0.146a ± 0.01

3.2. Fatty acids composition

Fatty acids composition is an important criterion for virgin olive oil. In fact, it is considered as a key for purity and authentication control. In addition, the high oxidative stability of virgin olive oil is related to its high monounsaturated/polyunsaturated ratio. Results (Table 2) showed that olive oil was characterized by low linoleic and palmitic acids content, ranging from 13.39 % to 15.62 % and from 13.77 % to 16.2 %, respectively, for Chemleli and Ouesleti varieties. Oleic acid was also present with high proportions of 62.5 % and 65.64 %, respectively, for Chemleli and Ouesleti varieties. Those findings are in agreement with data from literature [17][18]. The results demonstrated that olive oil is mainly a source of oleic acid (> 70% of total Fatty acids) and contains a low content of linoleic acid compared to the other vegetable oils. All samples were characterized by a high content of monounsaturated fatty acids and a balanced content of polyunsaturated fatty. Monounsaturated fatty acids were preferred by the health conscious. Similar results were found by Abazza et al. [19]. Results showed similarity between fatty acids composition of samples from Chemlali and Oueslati varieties but these latter were significantly ($P<0.05$) richer in oleic acid. However, samples from Chemlali variety were richer in saturated fatty acids especially palmitic and linoleic ones. This is probably related to genetic feature of these varieties and to the geographical growing area. Similar results revealing a relation between fatty acid composition of olive oil and cultivars, was found by Mannina et al. [20] who studied olive oil in a well-limited geographical region, with no consideration of the pedoclimatic factor (soil characteristics such as temperature and humidity).

Table 2: Fatty acids composition as determined by Gas Chromatography (% m/m methyl esters)

	Olive oil samples	
Fatty acids	**Oueslati variety**	**Chemlali variety**
C16	$14.51^{b} \pm 0.68$	$16.43^{a} \pm 0.69$
C16:1	$1.54^{b} \pm 0.38$	$2.22^{a} \pm 0.19$
C17	$0.03^{b} \pm 0.01$	$0.04^{a} \pm 0.02$
C17:1	$0.07^{b} \pm 0.02$	$0.10^{a} \pm 0.10$
C18	$2.03^{a} \pm 0.26$	$2.13^{a} \pm 0.15$
C18:1	$65.64^{a} \pm 1.55$	$62.11^{b} \pm 1.88$
C18:2	$14.81^{b} \pm 0.83$	$15.67^{a} \pm 1.21$
C18:3	$0.65^{a} \pm 0.08$	$0.66^{a} \pm 0.06$
C20	$0.42^{a} \pm 0.06$	$0.38^{b} \pm 0.03$
C20:1	$0.28^{a} \pm 0.05$	$0.26^{ab} \pm 0.06$

3.3. Chlorophylls and carotenoids contents

Chlorophyll and carotenoid pigments greatly influences the colour of Virgin Olive Oil. In this study, olives picked early in the season tend to make green colored oil as they contain high level of chlorophylls. However, olives harvested late in the season will typically produce more golden colored oils due to a higher level of natural carotenoids. Fig. 1 showed oil chlorophylls and carotenoids contents. Results demonstrated that oil from Oueslati variety was significantly ($P<0.05$) richer in carotenoids (1.74 mg/kg) and chlorophylls (2.53 mg/ kg) than samples from Chemleli variety (1.63 mg/kg and 2.29 mg/kg, respectively). In the other several studies the pigment content is higher than our results .Indeed chlorophyll content ranged from 5.5 to 9.5 and the carotenes content ranged from 3.00 to 4.9 depending on the variety [21]. Moreover, several studies showed that pigment amount is independent of the olive variety and the time of picking [22]. Since all olives were picked in the same time, the richness of Oueslati samples is very interesting for their oxidative stability and promotes this variety to be protected and valorized. In fact, chlorophylls and carotenoids were considered as bioactive substances and contribute to olive oil color. Both groups of compounds have functional properties because they affect the oxidative stability of olive oil [23].

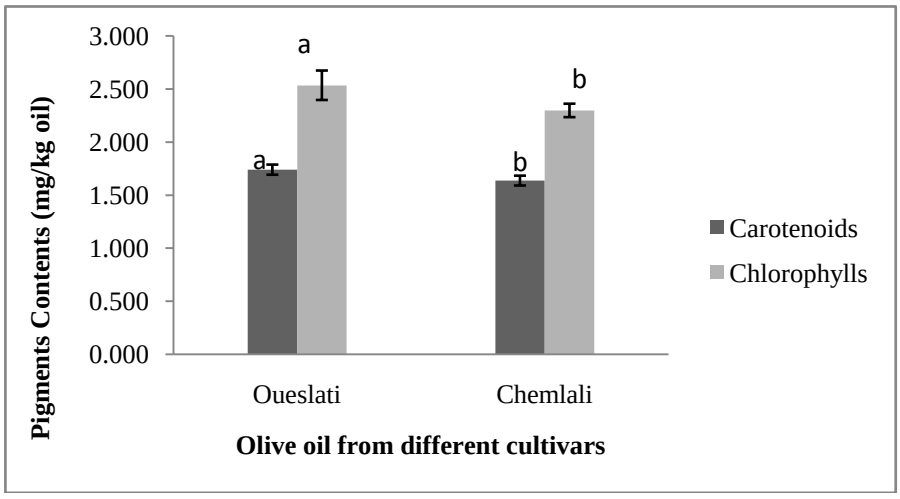

Figure 1: Pigments content of olive oil (mg/kg of olive oil)

3.4. Tocopherols contents

α-tocopherol is considered as the major antioxidant of virgin olive oil [24]. Several works showed the antioxidant effect of α-tocopherol on lipids [25]. Significantly ($P<0.05$) higher amounts of α-tocopherol were observed in most of the samples from Oueslati variety ranging from 156 to 252 mg/kg comparing to those from Chemlali ²one (100 – 188 mg/kg). These contents are generally in agreement with data previously reported for virgin olive oil ranging from 90 to 300 mg/kg [26] [27]. Also some works have reported a relationship of the tocopherols contents with the geographic origin of the olives. [28]These results permitted to conclude that oil from Oueslati variety was characterized by higher oxidative stability then that from Chemlali one.

3.5. Phenolic contents

Phenols from olive fight various reactive oxygen species and also counter act the damage caused by free radicals to cells (lipid peroxidation) [29]. The good correlation between oxidative stability and phenols content of the oil has been established by many several studies. [30,31] Results presented in Fig 2, showed that oils extracted from Oueslati variety contained significantly ($P<0.05$) higher phenolic contents ranging from 200 mg/kg to 350 mg/kg than comparing to those extracted from Chemlali variety (100 mg/kg to 200 mg/kg). This difference was probably related to genetic feature of each variety since their growth is in the same climatic and geographical conditions and the extraction was carried out with the same solvent. Similar results were found by Abouzar et al. [30]. Indeed, phenols content of oils samples ranged from 150 at 180 mg/kg. It seems through that olive polyphenols depend on the cultivar. Furthermore, several studies showed that oive polyphenols depend both on the cultivar and the origin area, without overlooking pedoclimatic conditions [32]. Indeed Ouini et al.[16] reported that oils from Oueslati variety had a phenols content of 528 mg / kg. The richness of samples from Oueslati variety in antioxidant substances offers to this variety a very interesting specificity.These results are with concordance with many studies that showed that oil from Oueslati variety was characterised by a good content of total phenols, tocopherols and a good resistance to oxidation. [33]

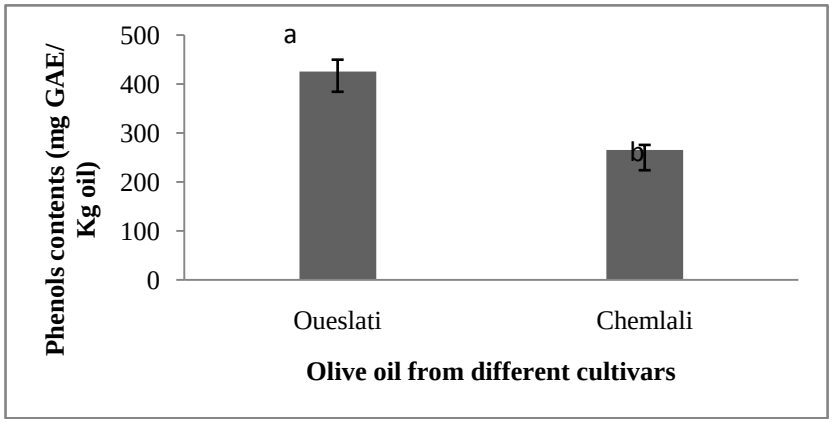

Figure 2: Phenols content of olive oil (mg GAE/kg of oil)

3.6 Sensory analysis

A large increase in demand for virgin olive oil of good quality is due not only to its health virtues but also to its organoleptic properties [34]. Sensory profiles of oil samples were represented in Tables 3 and 4. A large variability in oil sensory characteristics, according to geographical growing areas, was noted. As they are made from greener olives, oils from Oueslati variety, were generally more bitter, more pungent and more grassy/herbaceous in aroma and flavour than that extracted from Chemlali variety grown in the same geographical area but in Essbika province (fig.3)(table 4). Furthermore, oils from Oueslati variety were very specific. Indeed results presented in Table 3 noted that these samples had a significantly ($p>0.05$) higher intensity of the sensory characteristic fresh almond. Moreover, results showed that these samples were sweet and harmonious. This sensation is produced by the perception of the product components as olfactory, gustatory, tactile and kinesthetic

stimuli because they are present in suitable concentration ratios. Sensory profile of oils from Oueslati variety revealed that the perception of different notes and aromas was the result of the synergic effect of the oils' components, whose composition was influenced by the geographical growing area. [35]

Table 3: Main descriptors of fruity oil from Oueslati variety

Olive variety	**First descriptor**	**Second descriptor**	Additional descriptors
Oueslati variety	almond	Apple	Floral /olive leaf/grass
Chemlali variety	Leaf / grass	Floral	-

Table 4: Main descriptors intensity of olive oil samples

Olive oil samples	**Fruity intensity**	**Bitter intensity**	**Piquant intensity**
Oueslati variety	4,575[b]	3,107[a]	2,331[a]
Chemlali variety	3,269[a]	1,388[b]	1,637[b]

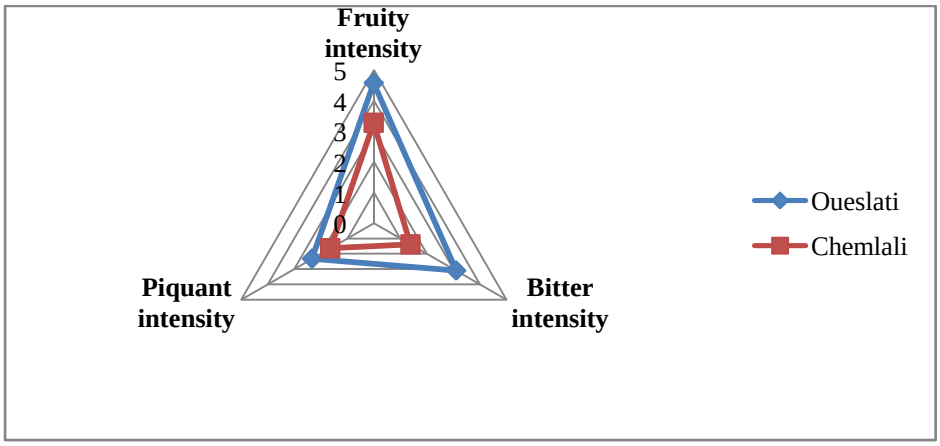

Figure 3: Sensory profile of olive oil

Conclusion

The main goal of this work was to characterize oils belonging to the region of Kairouan in order to install a Protected Geographical Identification (PGI). Indeed, the geographical specificity of this area, its protection capacity and olives varieties quality requires protection and also valorization. The study permitted concluded that the cultivar Oueslati was characterized by a very specific organoleptic profile compared to the Chemlali variety. Indeed, oils from Oueslati variety were sweet, harmonious and had an almond taste. In addition, these oils were characterized by higher oxidative stability and antioxidant content .These results are very interesting to install PGI for oil from "Oueslati" variety growing in Alaa province.

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References

1. García-González D.L., Aparicio R., *European Journal of Lipid Science and Technology* 110 (2008) 602–607.
2. Montealgre C., Algre M, Garcia-Ruiz M.L., *Journal of Agricultural and Food Chemistry* 58 (2010) 28-38.
3. Javier S., Rosana P., Moruno C., Ruiz-Gutierrez V., *The Journal of Nutritional Biochemistry* 17 (2006) 429.
4. Covas MI., *Pharmacological Research* 55 (2007) 175-186
5. Fuentes F., Lopez-Miranda J., Perez-Martinez P., Jimenez C., Marin Y., Gomez P., *Br. J. Nutr.* 100 (2008) 159.
6. Besnard G., Bervillé A., *Comptes Rendus de l'Académie des Sciences - Series III - Sciences de la Vie* 323 (2000) 173-18
7. Belaj A., Muñoz-Diez C., Baldoni L., Satovic Z., Barranco D., *Scientia Horticulturae* 124 (2010) 323–330
8. Belaj A., León L., Satovic Z., De la Rosa R., *Scientia Horticulturae* 129 (2011) 561-569
9 Hannachi H., Breton C., Msallem M., Ben El Hadj S., El Gazzah M., Bervillé A., *Scientia Horticulturae* 116 (2008) 280-290
10. European Community, Commission Regulation 640/2008, (2008). Amending Regulation No.2568/91/EEC. Official Journal of the European Communities, July 4, L178, 11-16.
11. Conseil Oléicole International COI/T.20/Doc. nº 24 (2001)
12. Minguez-Mosquera M.I., Rejano L., Gandul B., Sanchez H.A., Garrido J., *J. Am. Oil Chem.* Soc 68 (1991) 332-336
13. ISO 9936 (2006) Détermination des teneurs en tocophérols et en tocotriénols par chromatographie en phase liquide à haute performance.
14. Conseil Oléicole International Determination of biophenols in olive oils by HPLC. COI/T.20/Doc n°. 29 (2009)
15. Conseil Oléicole International COI/T.20/DOC.15 (2015).
16. Ouni Y., Flamini G ., Douja D., Zarrouk M., *Journal of Horticulture and Forestry* 3 (2011) 13-20 .
17. Nathalie V., Marine O., Laure D. , Jayne I., Martial L., *OCL* 17 (2010) 185-192.
18. Elkacmi R., Kamil N., Boulmal N., Bennajah M., *J. Mater. Environ. Sci.* (2016) 1485-1494.
19. Abaza L., Daoud D., Msallem M., Zarrouk M ., *Oléagineux Corps gras Lipides* 9 (2001)174–179.
20. Mannina L., Dugo G., Salvo F., Cicero L. , Ansanelli G., Calcagn C., Segre A., *J. Agric. Food Chem.* 51 (2003) 120–127
21. Alam Z., Chemistry and Physics of Lipids, 165 (2012) 277-281
22. Cerretani L., Motilva M.J., Romero M.P., Bendini A., Lercker G., *Eur. Food Res. Technol* 226 (2008) 1251
23. Chen H., Angiuli M., Ferrari C., Tombari E., Salvetti G., *Food Chemistry* 125 (2011) 1423-1429
24. Esterbauer H., Dieber-Rotheneder M., Striegl G., & Waeg G., *Am. J. Clin. Nutr.* 53 (1991) 314–321
25. Gutierrez F., Jiménez B., Ruiz A., Albi MA., *J Agric. Food Chem.* 47 (1999) 121-127
26. Deiana M., Rosa A., Cao C.F., Pirisi F.M., Bandino G., Dessi M.A., *Journal of Agricultural and Food Chemistry* 50 (2002) 4342–4346
27. Aguilera M.P., Beltran,G., Ortega, D., Fernandez, A., Jimenez, A., Uceda M., *Food Chem* 89 (2005) 387-391
28. Esti M., Cinquanta L., La Notte E., *Journal of Agriculture and Food Chemistry* 46 (1998) 32–35
29. Nick K., Maria Z. T., Antioxidants 3 (2014) 387–413.
30. Abouzar H., Reza F G. , 1 Davood B., Samaneh A S., AJCS 4 (2010) 258-263.
31. El Ouadi Y., Amirou A., Bouyanzer A., Elmsellem H., Majidi L., Bouhtit F., Hammouti B., *Maghr. J. Pure & Appl.* 1 (2015) 18-24.
32. Temime S.B., Wael T., Bechir B., Leila A., Douja A., Mokhtar Z., J. Food Lipids 13 (2006) 88–99
33. El Riachy M., Priego-Capote F., Leon L., Rallo L., de Castro M.D.L., *Eur. J. Lipid Sci. Technol* 113 (2011) 692–707
34.Tovar M.J., Romero M.P., Alegre S., Girona J., *J. Sci. Food & Agric.* 82 (2002) 1755-1763
35. Cecchi T., Passamonti P., Alfei B., Cecchi P., *International Journal of Food Properties* 14 (2011) 483-495.