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Original Article

EFFECTS OF OLIVE DRYING AND STORAGE ON THE OXIDATIVE STATUS, AROMA, CHLOROPHYLL AND FATTY ACIDS COMPOSITION OF OLIVE OIL

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ABSTRACT

Objective: In this study, we thoroughly investigate the effect of drying and storage of olives from four Tunisian cultivars (Chetoui, Chemlali, Oueslati and Picholine) on the final composition of olive oil.

Methods: Olives were dried using three different methods: ambient air, infrared radiation and oven heating. Oven-dried olives were stored during six months. Extraction was conducted using a soxhlet apparatus. Its quality was assessed by analyzing the fatty acid and aroma composition on one hand, and on the other hand by evaluating the total chlorophyll content and measuring specific extinctions at 232 and 270 nm.

Results: The main results show that air dried fruits (Chetoui cultivar) gave the most pigmented oil (3.32 ppm of total chlorophylls) followed by oven dried olives (1.12 ppm), whereas infra-red dried olives had the least amount of chlorophylls (0.98 ppm). Furthermore, the highest amount of total aroma was found in oven dried fruits whereas the lowest one characterized infrared dried olives. Fatty acid composition of our oils wasn't affected by drying techniques. Also, oven dried and stored olives showed an insignificant change in chlorophyll contents and aroma composition (Chetoui variety), coupled with a decreased level of total fatty acid amount as of the third month of preservation.

Conclusion: Drying techniques and storage affected aroma compounds, while oil oxidation, chlorophyll and fatty acid composition were unaffected. A better control for drying and storage should be developed to insure a better quality of olive oil. A comparison should be done between the current study and salt and dried olive preservation in order to offer hypertensive patients fruits with preserved nutritional values and the peculiar delicate flavour characteristic of olive oil

K**eywords:** Olive drying, Storage, Fatty acids, Pigments, Aroma.

INTRODUCTION

The olive agri-food chain constitutes the largest agro-industrial sector in the Mediterranean basin [1]. Since prehistoric times, olive products, mainly table olives and extra virgin olive oils (EVOO), have always been considered a gastronomic delicacy [2]. table olives (TO) and extra virgin olive oils (EVOO), both typical constituents of the Mediterranean diet, are rich in unsaturated fatty acids (FA), pigments and aroma. Olive products owe their preventive reputation against cancer and cardiovascular diseases to their balanced composition in FA and other minor components that are particularly antioxidants [3-5].

Olive products are also lauded for their flavour which constitutes another important quality criterion. A mixture of volatile compounds including aldehyde, ketones, alcohols, and esters generates a balanced flavour of green and fruity sensory characteristics, giving rise to pleasant gustatory and olfactory sensations that are very appreciated by consumers [6-9].

The organoleptic properties of olive products combined with their proven health benefits [10] along with the increase in their consumption stress the need for new and improved technologies focusing on good quality and preservation of olives and olive oil. While table olives can be prepared by either traditional methods or new technological processing involving blanching, salting and drying of mature black olives; a new technological procedure, involving drying of olives, is detailed hereafter. Usually, extraction procedure follows generally drying of raw material, and is used to reduce the moisture content and avoid the interference of water. The drying process may however affect both the matrix structure and the bioactive components [11-12]. Nonetheless, the drying of Olives was proven to be quite effective in reducing olive oil pigments, as well as affecting the composition of fatty acids and other components. In addition, drying techniques affect other quality parameters, such as oxidative stability and aroma volatile composition [13].

Currently, fruit drying is commercially widely employed in several countries for disease and quality control throughout processing, and chilling storage applications. Fruit exposure to moderate temperatures often increases storage life and affects the colour of the fruit. It was reported that pigment amounts vary with the species, the temperature and some other conditions [14].

In addition, classical preservation techniques based on pickling are not suitable for all consumers, especially those suffering from hypertension. The purpose of the present study was to ascertain the effects of different drying methods on FA composition, total aroma and total chlorophyll amounts, as well as to document the quality changes of the oven dried and stored olive fruits, in an attempt to develop a product that is suitable for consumption by hypertensive patients that are subject to a salt-free diet.

MATERIALS AND METHODS

Material

Olive fruits belonging to Picholine, Chemlali, Oueslati and Chetoui cultivars were harvested manually at the same ripening stage. Chetoui, Chemlali and Picholine cultivars were grown in the region of Mornag in the North-East of Tunisia, whereas Oueslati olives were picked up at the region of El Kef (North-West of Tunisia).

Methods

Drying techniques

- Three different drying methods were used to create a stock of dried olives from each different cultivar. Thus, three different dried olive stocks per cultivar were stored in dark glass bottles for 6 months at room temperature. Three sample replicates from each stock relative to each drying method, were assayed every 15 days during the period of 6 months for aroma, FA and chlorophyll measurements. Dried olives were stored the constituents of each sample were randomly gathered from corresponding dried fruit stock to obtain a final weight of 0.1 kg for each sample The three different drying techniques are as follows:
- Drying at ambient air (24°C ±4°C),
- Drying by infrared radiation (model, type, country) at 40°C.
- Oven-drying (model, type, country) at 40°C. A 40°C temperature were adopted because it allows the modulation of different quality parameters. The selection of a 40°C temperature was adapted to due to its effectiveness in the modulation of different quality parameters.

Table 1: Corresponding date of olive storage

Symbol	*D1*	*D2*	*D3*	*D4*	*D5*	*D6*	*D7*	*D8*	*D9*	*D10*	*D11*	*D12*	D13
Days of preservation	1	15	30	45	60	75	90	105	120	135	150	165	180

Oil extraction

Oil extraction for all samples was performed using a Soxhlet apparatus.

Total chlorophyll content

Chlorophyll contents in olive oil samples were determined by measuring the absorbance at 630 nm, 670 nm, and 710 nm. Carbon tetrachloride served as control. This approach was based on the standardized method set forth by the American Oil Chemists Society (AOCS).

Specific extinctions

Several methods have been used to assess the oxidative status of lipids, based on the detection of primary or secondary oxidation products and the analysis of the oxidation substrate [15]. According to the European Commission (EC) regulation [16], the oxidative deterioration in olive oils is assessed by measuring the absorbance at 232 nm and 270 nm to evaluate conjugated dienes and trienes resulting from primary and secondary oxidation products.

Fatty acids methylation and analysis

Fatty acids were converted into their methyl esters (FAMEs) before analysis according to the method described by [17]. Then, FAMEs were analysed by gas chromatography using an HP 6890 chromatograph equipped with a flame ionization detector (FID), an EPC (Electronic Pressure Control) injector and an Innowax capillary column (HP Innowax, Agilent Technologies, USA) (30m, 0.25 mm i. d., 0.25 µm film thickness) with a polar stationary phase made of polyethylene glycol (PEG). The carrier gas was N2 (U). The flow rate was 1.5 ml/min and the split ratio 100:1. The detector and injector temperatures were held 275°C and 250°C, respectively. The oven temperature was programmed as follows: isotherm at 150°C for 1 min; increase in temperatures from 150 to 200°C at a rate of 15°C/min; isotherm at 200°C for 3 min and then another increase in temperatures from 200 to 242°C at a rate of 2°C/min. FA composition was determined as a percentage of the total fatty acids (TFA).

Aroma extraction and analysis

Fifty grams of oil were put into 120 ml Drechel gas washing bottle equipped with a porous distributor. Volatiles were stripped with nitrogen (1.2dm^3 min^{-1}, 37°C) for 2 hours, trapped on 50 mg of activated charcoal purchased from E. Merck (Schuchardt, Germany) (0.5–0.85 mm, 20–35 mesh ASTM) with N2 for 120 min and eluted with 1 ml of diethyl ether. The extract was concentrated using a Vigreux column at 35°C until a final volume of about 50 µl is obtained [9]. GC analyses were performed with the same apparatus and column previously described using the following conditions. The carrier gas was N2 (U) whereas the flow rate and the split ratio were set to1.6 ml/min, and 60:1 respectively. The injector and the detector temperatures were respectively set to 250 and 300°C. The oven temperature was programmed as follows: an isotherm at 35°C for 10 min; an increase in temperatures from 35°C to 205°C at a rate of 3°C/min and an isotherm at 205°C for 10mn.

Statistical analysis

The results are expressed as mean values of three replicates ± standard deviation. The mathematical procedure was carried out using statsoft statistica software package on a Pentium computer.

RESULTS AND DISCUSSION

Fatty acids composition

Dehydration of biological material is a controlled effort to preserve the structure or create a new product that serves a functional purpose. In this context, food dehydration is revisited from the perspective of recent advances in food material science [18]. The time required for olives drying depends on their kind and their size, their moisture content, the humidity of the air during the process and the efficiency of the dehydrator. Reducing the amount of salt used in olives during olive processing and preservation seems to be an important innovation though this is not a straightforward process, as salt plays an important role in the microbiological quality of the end product [19]. Among all the analyzed control oil samples, the most abundant FA was the oleic acid that ranged from 55.9 % to 65.4 % of TFA (Table 2). “Chetoui” oil had the highest level of oleic acid (65.4 % of TFA), compared to “Chemlali” (55.9 % of TFA). The highest level of palmitic acid was found in “Chemlali” oil (16.9 % of TFA) whereas the lowest level was in “Chetoui” (10.8% of TFA). Our results show that the palmitoleic and stearic acids proportions ranged from 1.0% to 3.0% and 1.8% to 3.3% of TFA, respectively.

Table 2: Percentage of Fatty acids composition for the four cultivars

Fatty acids %	Norm (COI)	Current study			
		Chetoui	Picholine	Chemlali	Oueslati
Palmitic acid (C16: 0)	7.5-20.0	10.8 ± 0.1^{c}	10.8 ± 0.1^{c}	16.9 ± 0.1^{c}	12.4 ± 0.3^{c}
Palmitoleic acid (C16: 1)	0.3-3.5	0.7 ± 0.0^{f}	1.0 ± 0.3^{f}	3.0 ± 0.3^{d}	1.6 ± 0.2^{e}
Stearic acid (C18: 0)	0.5-5.0	3.3 ± 0.0^{d}	1.8 ± 0.03^{d}	2.4 ± 0.3^{d}	2.2 ± 0.1^{d}
Oleic acid (C18: 1)	56.0-83.0	67.2 ± 0.0^{a}	65.4 ± 0.0^{a}	55.9 ± 0.2^{a}	63.4 ± 0.1^{a}
Linoleic acid (C18: 2)	3.5-20.0	16.2 ± 0.1^{b}	19.2 ± 0.1^{b}	19.1 ± 0.2^{b}	18.6 ± 0.2^{b}
Linolenic acid (C18: 3)	0.0-1.5	1.5 ± 0.1^{e}	1.4 ± 0.2^{e}	1.1 ± 0.2^{f}	1.5 ± 0.1^{e}

FA composition of "Picholine", “Chemlali”, "Oueslati" and "Chetoui" oils before and after drying by different techniques are presented in table 3. This composition remained practically invariable after drying. The major FA was the oleic acid (67.09% to 68.8% of TFA), followed by palmitic (10.38% to 10.47% of TFA), linoleic (15.68% to 17.30 % of TFA), linolenic (1.07% to 1.53% of TFA), stearic (1.8% to 3.3% of TFA) and palmitoleic acids (0.56% to 0.84% of TFA).

Table 3: Changes of fatty acids composition and effect of drying techniques on the different cultivars

		C16:0	C16:1	C18:0	C18:1	C18:2	C18:3	Saturated FA	Unsaturated FA
Control	Chetoui	10.8±0.1^{g}	0.7±0.3^{d}	3.3±0.2^{a}	67.2±0.1^{c}	16.2±0.1^{g}	1.5±0.1	14.2±0.2^{e}	85.8±0.3^{a}
	Chemleli	16.9±0.1^{a}	3.0±0.3^{a}	2.4±0.3^{bc}	55.9±0.2^{l}	19.1±0.2^{b}	1.1±0.2	19.3±0.1^{a}	80.7±0.1^{d}
	Picholine	10.8±0.1^{g}	1.0±0.3^{d}	1.8±0.3^{c}	65.4±0.0^{g}	19.2±0.1^{b}	1.4±0.2	12.6±0.2^{f}	87.3±0.1^{a}
	Oueslati	12.4±0.3^{e}	1.6±0.2c^{d}	2.2±0.1^{c}	63.4±0.1^{k}	18.6±0.2^{cd}	1.5±0.1	14.6±0.1^{d}	85.4±0.1^{b}
Ambient air	Chetoui	10.43±0.1^{i}	0.82±0.2^{d}	3.2±0.1^{a}	68.45±0.1^{b}	15.49±0.3^{i}	1.53±0.1	13.66±0.3 ^{e}	86.33±0.1^{a}
20°C±4	Chemleli	16.45±0.1^{b}	2.45±0.3^{b}	2.0±0.3^{c}	56.3±0.2^{l}	18.81±0.2^{cd}	1.8±0.2	18.45±0.1^{b}	81.55±0.2^{c}
	Picholine	10.2±0.1^{j}	1.6±0.3^{cd}	2.1±0.3^{bc}	65.7±0.0^{d}	19.82±0.1^{a}	1.6±0.2	12.3±0.2^{f}	87.7±0.1^{a}
	Oueslati	11.9±0.3^{ef}	1.5±0.2^{cd}	2.3±0.1^{bc}	64.1±0.1^{h}	17.9±0.2^{e}	1.7±0.1	14.2±0.2^{e}	85.7±0.2^{b}
Oven 40°C	Chetoui	10.38±0.1^{i}	0.56±0.2^{d}	3.3±0.1^{a}	68.8±0^{a}	15.7±0.0^{h}	1.13±0.4	13.74±0.1^{e}	86.26±0.1^{a}
	Chemleli	15.8±0.1^{c}	2.9±0.3^{b}	2.6±0.3^{bc}	56.4±0.2^{l}	19.3±0.2^{b}	1.9±0.2	18.4±0.2^{b}	81.6±0.2^{c}
	Picholine	10.68±0.2^{g}	1.12±0.3^{d}	1.2±0.3^{d}	64.6±0.0^{f}	18.9±0.1^{c}	1.7±0.2	11.88±0.1^{g}	88.12±0.3^{a}
	Oueslati	12.34±0.3^{e}	1.36±0.2^{d}	2.5±0.1^{b}	63.54±0.1^{j}	18.46±0.2^{d}	1.65±0.1	14.85±0.2^{d}	85.15±0.3^{b}
Infra-red 40°C	Chetoui	10.47±0.1^{h}	0.59±0.1^{d}	3.4±0.1^{a}	67.0±0.1^{c}	17.3±0.3^{f}	1.07±0.4	13.94±0.1^{e}	86.06±0.4^{a}
	Chemleli	15.29±0.1^{d}	2.4±0.3^{b}	2.3±0.3^{bc}	55.45±0.2^{l}	19.5±0.2^{ab}	1.1±0.2	17.59±0.2^{c}	82.51±0.1^{c}
	Picholine	10.1±0.1^{j}	1.7±0.3^{cd}	1.6±0.3^{c}	65.6±0.0^{e}	18.9±0.1^{c}	1.7±0.2	11.7±0.1^{h}	88.3±0.2^{a}
	Oueslati	11.7±0.3^{f}	1.8±0.2^{c}	2.1±0.1^{b}	63.8±0.1^{i}	18.4±0.2^{d}	1.9±0.1	13.8±0.2^{e}	86.2±0.2^{a}

The oleic acid percentage was not affected by the drying temperatures. In all drying techniques, the percentage of total unsaturated fatty acids (TUFA) in dried olives was almost the same as in fresh ones.

As for oven-dried Chetoui olives, table 3 shows the abundance of different UFA (85.8%) such as C18:1 and C18:2 whose rates accounted for 67.2% and 16.2% of TFA, respectively. C16:1 was encountered in traces. Saturated fatty acids (SFA) accounted for 14.2% of TFA and are represented mainly by palmitic (C16:0) (10.8%) and stearic (C18:0) (3.3%) acids.

For stored dried olives, FA composition was modified (Table 4) with a steady reduction of C18: one single content was observed as of the third month of storage and after 6 months its percentage decreased to 59.8% of TFA. This decrease of C18:1 percentage could be attributed to enzymatic activities that favour lipolysis due to a microbial lipolytic activity in addition to a spontaneous oxidization phenomenon. Oil FA composition does not depend on the duration of olive storage before the extraction [20].

Palmitic acid amount increased by 4% towards the end of storage, whereas percentages of palmitoleic and stearic acids remained practically unchanged. Most enzymatic reactions are closely conditioned by the activity of water. In various cases of hydrolysis reactions, enzymatic activity begins to appear when the activity of water is 0.2 even though some enzymes, such as lipases, represent an exception since their action can be observed at very low water activities [21].

The explanation of this behaviour resides in the fact that the substratum enzyme contact doesn't need an aqueous phase as vector because lipids are not miscible with water and lipases act at the interface oil-water.

Table 4: Variations in fatty acids composition of oven dried and stored olives

	C16:0	C16:1	C18:0	C18:1	C18:2	C18:3
Control (oven dried and non preserved fruits)	10.79±0.1	1.02±0.2	1.819±0.1	65.43±0.2	20.08±0.3	1.66±0.0
D1	10.79±0.1^{e}	1.02±0.2^{e}	1.81±0.5^{e}	65.43±0.2^{a}	20.08±0.3^{a}	1.66±0.3
D2	10.58±0.1^{e}	1.03±0.2^{e}	1.71±0.1^{e}	65.31±0.2^{a}	20.2±0.0^{a}	1.53±0.2^{d}
D3	10.05±0.3f	1.09±0.0^{e}	2.87±0.2^{c}	64.2±0.1^{b}	19.56±0.1^{b}	1.48±0.0^{d}
D4	9.85±0.1^{g}	1.21±0.3^{d}	2.68±0.2^{c}	62.58±0.1^{d}	20.15±0.1^{a}	2.59±0.3^{b}
D5	10.02±0.2^{f}	1.97±0.1^{c}	2.03±0.3^{d}	63.12±0.1^{c}	19.78±0.1^{b}	3.12±0.3^{a}
D6	8.25±0.2^{g}	1.86±0.1^{c}	3.02±0.2^{b}	65.12±0.1^{a}	20.3±0.3^{a}	2.16±0.2^{b}
D7	11.23±0.2^{d}	2.15±0.0^{b}	1.2±0.2^{f}	65.23±0.1^{a}	18.12±0.1^{c}	2.03±0.2^{c}
D8	10.54±0.5	1.02±0.1^{e}	2.05±0.1^{d}	64.53±0.3^{b}	20.08±0.2^{a}	1.6±0.3^{d}
D9	11.29±0.3^{d}	2.03±0.3^{b}	2.86±0.3^{b}	63.15±0.4^{c}	18.24±0.3^{c}	2.63±0.0^{b}
D10	12.89±0.3^{b}	2.05±0.2^{b}	1.98±0.1^{e}	63.18±0.4^{c}	16.25±0.3^{e}	3.12±0.1^{a}
D11	12.25±0.1^{c}	2.13±0.2^{b}	3.36±0.1^{a}	61.54±0.1^{d}	17.3±0.5^{d}	3.65±0.1^{a}
D12	14.35±0.1^{a}	3.31±0.3^{a}	3.05±0.1^{b}	59.78±0.2^{e}	16.24±0.5^{e}	3.25±0.3^{a}
D13	14.1±0.2^{ab}	3.34±0.3^{a}	3.13±0.1^{b}	59.88±0.3^{e}	16.23±0.5^{e}	3.15±0.0^{a}

Total pigments

Water, being the main component of foods, has a direct and decisive influence on their quality and shelf life through its effect on several physicochemical and biological changes. The total chlorophyll level is an important quality parameter of olive oil because it is correlated to the color which is a basic attribute for evaluating its organoleptic quality. Olive oil has a yellow-green to gold colour depending on its pigments content [22]. Chlorophylls are involved in oil auto-oxidation and photo-oxidation [23, 24].

Table 5 shows that total chlorophyll levels ranging from 2.09 ppm in oil extracted from fresh Chetoui olives to 0.86 ppm in Picholine. It varied from 3.32 ppm in oil extracted from Chetoui olives ambient air dried to 0.45 ppm in picholine olives which were infra-red dried. The total chlorophyll amount varied from 2.09 to 0.86 in fresh olives and from 3.32 to 0.45 in dried ones (Table 5). In particular, Chetoui had the highest level of chlorophylls, while the lowest level was observed in Picholine oil.

A significant decrease in pigment content was found when fruits were dried by infrared radiation (46 %) and by oven drying (54 %). Hence, air dried olives resulted in the most pigmented oil, which could be the result of fruit dehydration facilitating the migration of these pigments out of pulp cells. We can conclude that pigment levels in olive oil were influenced by the drying method. The drying by heating affected the total amount of chlorophyll that decreased significantly.

Table 5: Changes in olive total chlorophyll amount (ppm) after drying

	Control	Ambient air	Oven room 40°C	Infra-red radiation
Chetoui	2.09±0.03[a]	3.32±0.00[a]	1.12±0.1[a]	0.98±0.02[a]
Chemleli	1.92±0.12[a]	3.04±0.02[b]	0.97±0.04[b]	0.75±0.08[b]
Oueslati	1.14±0.21[b]	2.78±0.01[c]	0.84±0.06[c]	0.49±0.0[c]
Picholine	0.86±0.02[c]	1.52±0.05[d]	0.63±0.0[d]	0.45±0.01[d]

Table 6: Changes of total chlorophylls contents during six months of preservation in opaque bottles

Date	*D1*	*D2*	*D3*	*D4*	*D5*	*D6*	*D7*	*D8*	*D9*	*D10*	*D11*	*D12*	D13
Chetoui (ppm)	1.12	1.09	1.1	1.11	1.11	1.1	1.09	1.1	1.05	1.09	1.1	1.1	1.09

During their study on the effects of on olive oil color, Lutein and β-carotene contents increased as a result of heat-treatments of Olives [10]. Lutein content increased at least 2.2-fold compared to control olive oil. They also reported a significant increase in the amount of chlorophyllic compounds, such as chlorophylls *a* and *b* and pheophytins *a* and *b*. For instance, chlorophyll *a* content increased in a range of 2.0 to 7.7-fold compared to the amount found in control olive oil. The temperature of fruits in the crusher is the only factor responsible for this effect and the involvement of LOX activity in pigment degradation could be considered. Herbs drying resulted in a degradation of their chlorophylls and therefore in a color change (result of microwave-convective) [25]. According to these authors, the changes were related to the herb species, and materials belonging to a single family were characterized by similar stability.

The microwave-convective drying of herbs is a proven as a technique that affords dried material of high quality, providing that the process parameters adopted in the latter study are applied [25]. Additionally, the short time of the process can fuel the incentive for the application of that method of drying on an industrial scale since it constitutes a decisive economic factor. Nevertheless, the degree of retention of biologically active compounds and the extent of color change in the course of drying are related to the herb species, emphasizing on the need to study a large array of materials belonging to various taxonomic groups. Chlorophyll retention in the dried coriander leaves decreased with the increase in temperature and was at a maximum when coriander leaves were dried at 45°C. This result was in accordance with that of Rocha et al. (1993) [26] dealing with basil drying. Obviously, 45°C or a lower temperature may be recommended as the temperature of air for drying coriander leaves as it gave the product with the highest chlorophyll content. The product quality in terms of chlorophyll and rehydration capacity was found to be most acceptable when coriander leaves, blanched in hot water at 80°C for 3 min, were dried at 45°C [27]. Pigment levels in the dried and stored olives remained the same, suggesting that they did not undergo any deterioration (Table 6). Chetoui samples presented 1.12 ppm of total chlorophyll at the first day of storage D1. At the end of the storage period, total chlorophyll amount was of 1.09 ppm (Table 6). This slight decrease was deemed to be insignificant. In fact, it could be related to the preservation in the darkness [28]for a better preservation of these pigments.

Aroma composition

The qualitative and quantitative composition of accumulation products strongly depends on the levels of enzymes involved in the lipoxygenase pathway and on their activities [29]. Moreover, the production of metabolites is related to the ripening degree and storage time of fruits [30-32].

Our preliminary results (Table 7) show that oil from fresh and ripe fruits presented high amounts of total aroma compounds derived from the LOX pathway, i. e. C6 compounds obtained from the cleavage of linolenic and linoleic acids 13-hydroperoxides due to the hydroperoxide lyase (HPL) activity and C5 compounds derived from the homolytic cleavage of linolenic acid 13-hydroperoxides, reported in Italian cultivars [32].

The four studied varieties showed a significant decrease in the total aroma amounts (Table 7) as well as qualitative changes. Trans-2-hexenal, which gives a typical “green note”, is the most abundant compound in olive pulp [33]. The data obtained in our preliminary study shows that there is a significant qualitative difference in the aroma composition between dried and fresh fruits of all cultivars. The comparative aroma composition of olive oil obtained from control and dried fruits showed that oil from dried fruits gave rise to a modification in the aroma composition of olive oil. In particular, drying promoted an increase in C6 amounts, especially esters and alcohols (Fig 1, 2, 3, 4) [34]. Microwave convection to dry basil leaves was proposed as an alternative technique [34]. According to these authors, microwave dried basil leaves had a larger retention of both volatile compounds and chlorophylls when compared to the leaves dried by traditional techniques.

Table 7: Percentage of aroma compounds in fresh and dried olive for the different cultivars

		Aldehydes		Alcohols			Esters		Acid
		Hexanal	Z-2-hexenal	Hexanol	Z-2-hexenol	E-3-hexenol	E-3-hexenyl acetate	Hexyl acetate	Acetic acid
Control	Chetoui	10.4±0.1[c]	40.4±0.1[h]	10.3±0.3[h]	18.7±0.3[h]	14.0±0.3[c]	3.3±0.3[b]	2.8±0.1[e]	0.2±0.3[e]
	Chemleli	11.4±0.1[b]	71.0±0.1[a]	5.5±0.6[i]	8.8±0.3[l]	1.3±0.1[i]	0.6±0.1[gh]	1.3±0.1[i]	0.0±0.0
	Picholine	6.8±0.1[e]	51.7±0.1[d]	13.3±0.1[h]	26.6±0.3[e]	1.2±0.1[i]	0.2±0.1[i]	0.1±0.0[l]	0.0±0.0
	Oueslati	10.1±0.6[c]	60.7±0.4[b]	16.3±0.1[e]	9.8±0.3[k]	2.1±0.1[h]	0.5±0.0[h]	0.2±0.0[k]	0.3±0.0e
Ambient air	Chetoui	5.2±0.2[f]	35.6±0.2	12.2±0.1[i]	26.8±0.3[e]	13.0±0.1[d]	3.5±0.3[b]	1.8±0.3[h]	1.9±0.3[a]
20°C±4	Chemleli	15.0±0.3[a]	55.4±0.2[c]	10.5±0.3[h]	13.8±0.3[j]	2.0±0.0[h]	0.7±0.0[g]	2.0±0.3[f]	0.5±0.0[d]
	Picholine	4.5±0.7	43.6±0.2[f]	15.6±0.1[f]	33.5±0.3[c]	1.2±0.3[i]	0.1±0.0[j]	0.4±0.0[j]	1.3±0.1
	Oueslati	5.7±0.3[f]	54.1±0.2[d]	15.8±0.1[f]	20.0±0.3[g]	2.2±0.3[h]	0.3±0.0[i]	0.0±0.0	1.9±0.3[a]
Oven 40°C	Chetoui	7.7±0.3[d]	31.9±0.1[k]	17.4±0.1[d]	20.9±0.3[f]	14.9±0.0[b]	3.7±0.3[b]	2.7±0.0[e]	0.8±0.1[bc]
	Chemleli	11.8±0.2[b]	57.7±0.1[c]	11.0±0.5[j]	13.5±0.3[j]	1.6±0.1[i]	0.5±0.1[h]	3.4±0.1[d]	0.4±0.1[de]
	Picholine	2.5±0.1[h]	33.2±0.3[j]	19.7±0.5[c]	37.3±0.3[b]	5.5±0.3[g]	0.1±0.[1]	1.2±0.1[g]	0.7±0.1[c]
	Oueslati	2.4±0.1[h]	53.7±0.1[e]	24.0±0.2[b]	7.5±0.0[m]	6.9±0.1[f]	1.3±0.1[f]	3.8±0.1[c]	0.3±0.0[e]
Infra-red 40°C	Chetoui	3.1±0.1[g]	18.6±0.3[l]	16.3±0.2[e]	30.1±0.3[d]	20.6±0.1[a]	5.5±0.1[a]	4.9±0.1[b]	1.1±0.3[b]
	Chemleli	7.1±0.1[d]	39.6±0.1[i]	11.7±0.2[j]	26.6±0.3[e]	5.6±0.1[g]	1.9±0.2[e]	6.3±0.2[a]	1.4±0.3[b]
	Picholine	1.2±0.0[i]	31.2±0.9[k]	13.8±0.1[g]	39.9±0.3[a]	9.3±0.3[e]	1.1±0.1[f]	2.8±0.2[e]	0.8±0.1[c]
	Oueslati	3.3±0.0[e]	41.0±0.3[g]	26.3±0.1[a]	16.4±0.3[i]	6.4±0.6[f]	2.8±0.1[d]	1.6±0.3[g]	2.1±0.3[a]

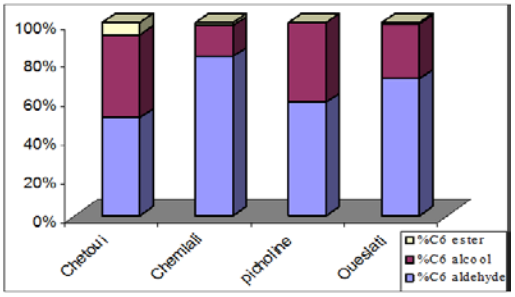

Fig. 1: Percentages of different aroma classes in control olive oils

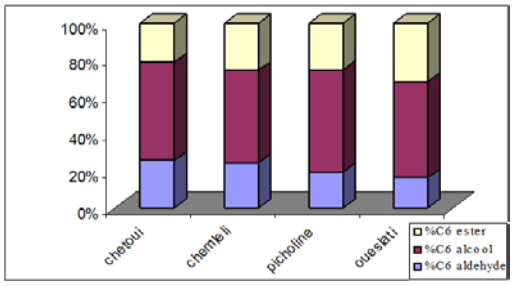

Fig. 2: Percentage of different aroma classes in oils extracted from oven dried olive

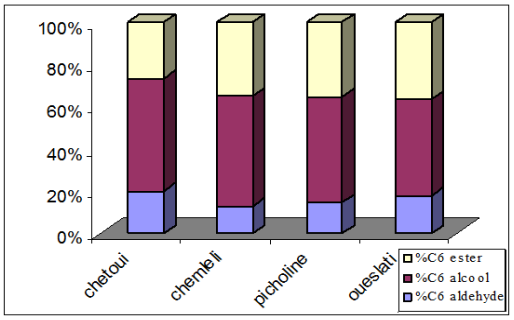

Fig. 3: Percentage of different aroma classes in oils extracted from infra-red dried olives

It is important to note that some aroma compounds products, during storage, result from sugars fermentation and amino acids transformation like ethanol which gives rise to sensory modification [35]. These compounds are supposed to be generated by different ways during olives storage.

Some aroma compounds contents decrease considerably during olives storage (Table 8). Hexanal and E-2-hexenal contents were in agreement with this finding, their composition out of total aroma compounds varied between fresh olives and dried olives at the last date of storage from 8.6% to 2.1% and 36.1% to 5.1% for hexanal and E-2-hexenal, respectively. Similarly, E-2-hexenol content decreased by 5%. Conversely, the hexanol and the Z-3-hexenol contents did not change after storage.

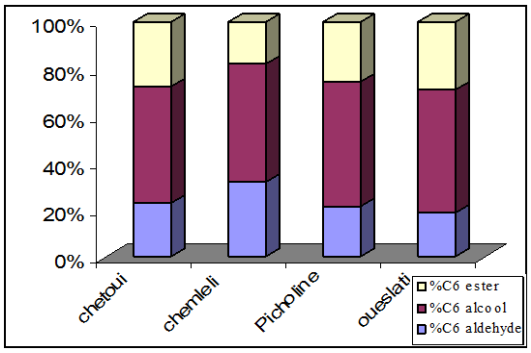

Fig. 4: Percentage of different aroma classes in oils extracted from ambient air dried olives

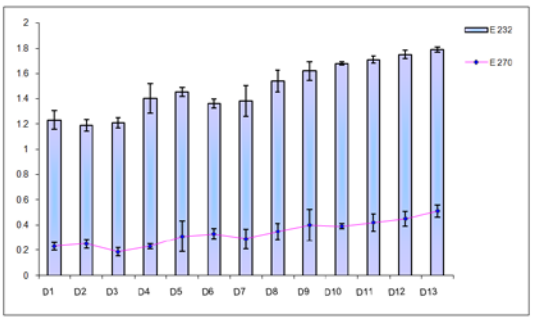

Fig. 5: Changes in specific extinction values of oven-dried Chetoui olives oil

Aldehydes percentages decreased meaningfully during the conservation, especially that of trans-2-hexenal. We also observed an increase of acids content such as acetic acid and esters such as hexyl acetate (Table 8). The two latter compounds are responsible for an unpleasant fragrance.

Table 8: Aroma compounds levels (%) of oven-dried and preserved olives (Chetoui variety)

	Hexanal	Z-2-hexenal	Hexanol	Z-2-hexenol	E-3-hexenol	E-3-hexenyl acetate	Hexyl acetate	Acetic acid
Control (oven dried and non preserved fruits)	8.66±0.3	36.10±0.3	19.68±0.3	23.64±0.3	16.8±0.3	4.2±0.3	3.1±0.3	0.9±0.3
D1	7.42±0.1^{b}	37.32±0.1^{a}	20.88±0.2^{a}	22.87±0.1^{b}	16.98±0.3^{e}	4.19±0.2^{c}	3.92±0.6^{c}	1.01±0.1^{i}
D2	8.09±0.1^{a}	31.67±0.1^{c}	19.65±0.2^{c}	23.09±0.1^{a}	16.02±0.1^{e}	4.87±0.2^{b}	3.67±0.4^{d}	0.87±0.1^{j}
D3	6.44±0.1^{c}	34.63±0.1^{b}	20.32±0.3^{b}	21.91±0.1^{c}	17.10±0.1^{d}	3.98±0.2^{cd}	3.03±0.1^{e}	2.23±0.2^{h}
D4	6.56±0.3^{c}	28.27±0.1^{d}	19.02±0.2^{d}	20.11±0.7^{e}	16.93±0.3^{d}	3.57±0.3^{e}	4.93±0.1^{b}	4.76±0.0^{f}
D5	4.89±0.3^{f}	28.22±0.1^{d}	18.61±0.2^{e}	21.18±0.5^{d}	16.28±0.1^{e}	5.11±0.1^{a}	4.89±0.1^{b}	4.43±0.3^{g}
D6	5.76±0.1^{d}	20.19±0.5^{e}	16.42±0.1	19.45±0.1^{e}	16.49±0.3^{d}	3.82±0.1^{d}	4.67±0.1^{c}	6.92±0.0^{d}
D7	5.14±0.1^{e}	20.11±0.3^{e}	18.23±0.1^{f}	19.82±0.1^{e}	17.33±0.1^{b}	2.28±0.1	5.83±0.1^{a}	6.01±0.2^{e}
D8	4.03±0.1^{g}	16.74±0.1^{f}	19.65±0.1^{c}	19.00±0.3^{f}	17.86±0.3^{a}	3.93±0.5^{de}	3.64±0.0^{d}	8.17±0.1^{c}
D9	4.81±0.5^{f}	12.28±0.1^{g}	18.18±0.1^{g}	17.19±0.3^{h}	16.27±0.2	3.44±0.5^{de}	3.75±0.3^{d}	6.81±0.3^{d}
D10	3.54±0.0^{i}	10.22±0.3^{h}	17.11±0.1^{h}	17.98±0.2^{g}	17.08±0.2^{c}	3.53±0.0^{e}	4.69±0.3^{c}	9.01±0.5^{b}
D11	3.89±0.2^{h}	6.99±0.3^{j}	18.17±0.1^{g}	17.28±0.2^{h}	17.67±0.2^{a}	3.09±0.1^{g}	4.75±0.0^{c}	9.18±0.3^{b}
D12	3.13±0.2^{j}	7.98±0.0^{i}	18.54±0.7^{e}	16.19±0.2^{i}	17.10±0.8^{c}	3.16±0.1^{g}	4.95±0.1^{b}	9.82±0.0^{a}
D13	2.13±0.2^{h}	5.16±0.3^{k}	18.97±0.1^{e}	17.24±0.2^{h}	17.1±0.3^{c}	3.18±0.3^{g}	4.89±0.3^{bc}	9.73±0.3^{b}

Air exposure of stored olive oils was correlated to an increase of the amount of some negative sensory components such as penten-3-ol and hexanal and a parallel decrease of that of compounds with positive attributes like *trans*-2-hexenal [36]. These changes would be expected to reduce quality [36].

Genesis of oxidation products

Reactions of oxidization in the plant oils are ineluctable and result from several phenomena influencing the stability such as auto-oxidation, photo-oxidation and thermo-oxidization.

Specific extinctions measurements of oils extracted from fresh, dried and dried/stored samples revealed the presence of oxidation products (Table 9). Specific extinctions increased in ambient air dried olives; this can be explained by a degradation of the unsaturated free FA (FFA) under the action of microbial enzymes. Oven-dried olives (40°C) showed the lowest extinction value in comparison with olives treated by other drying techniques; the infrared dried olives and the air dried fruits were characterized by the highest extinction values, implying a high oxidation level. The microwave heating process could accelerate oxidative reactions which promotes the involvement of free radicals [37].

Table 9: Specific extinctions effect of drying techniques in oils from four cultivars

		Control	Ambient air	Aven 40°C	IR 40°C
Chetoui	E 232	0.23	0.345	0.32	0.4
	E 270	1.23	1.49	1.26	1.35
Chemleli	E232	0.21	0.33	0.30	0.376
	E270	1.31a	1.57	1.23	1.43
Oueslati	E 232	0.22	0.37	0.35	0.32
	E 270	1.17	1.55	1.24	1.41
Picholine	E 232	0.24	0.51	0.363	0.38
	E 270	1.19	1.49	1.25	1.33

Several other factors could also interfere in lipid oxidation processes, such as FA composition [38], FFA, oxygen exposure [39], heat, water, light [40] and antioxidants [38] between others. The specific extinction evolutions of the oils from dried and stored olives are shown in Fig 5. We noticed a progressive increase of the specific extinctions during the storage, which proves their oil oxidation generating conjugated dienes that absorb at 270 nm. It is difficult to stop these oxidization reactions, but it is possible to limit them by lowering the water activity. The increase of the specific extinctions values could explain the oxidization phenomena intervention noticed in the analysis of FA composition of dried and preserved olives.

Air drying has been the main drying method following osmotic dehydration. MW or MW-convective drying of osmotically dehydrated products has been however shown in the literature to improve the drying rate and retain product quality compared to air drying [41].

CONCLUSION

- Volatile compounds analysis showed an increase in the rate of alcohols and esters versus a decrease of aldehydes independently of the drying method (oven drying at 40°C and infrared drying). Fatty acid composition remained however unchanged.

- Spectrophotometric analysis of oils showed that infra-red dried olives had the less pigmented oil, while the air-dried olives had the most pigmented oil. On the other hand, chlorophyll content of oven-dried olives decreased by a half. Oil specific extinction was the highest in infrared dried olives. Oven-dried/stored olives showed that qualitative changes in the aroma composition was due to an increase of hexyl acetate and acetic acid contents and a decrease in hexanal, Z-2-hexenal, hexanol, Z-2-hexenol, E-3-hexenol contents.

The percentages of oleic and linoleic acids decreased by 4% while palmitic acid increased. The composition of chlorophyll was stable during storage. Gradual increase in the values of specific extinctions in oil from dried olives was observed during storage. It seemed as though drying techniques and storage affected aroma compounds, while oil oxidation, chlorophyll and fatty acid composition were unaffected. It would be thus useful to be able to develop a better control for drying and storage to insure a better quality of olive oil. This study can go forward by investigating the difference between salt and dried olive preservation to prove the efficacy of our results, evaluating other parameters such as phenols and β-carotene and studying the correlation between aroma composition and flavour in order to offer hypertensive patients fruits with preserved nutritional values and delicate flavour highly appreciated by consumers.

CONFLICT OF INTERESTS

Declared None.

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