Variation of the Fatty Acid Composition of Olive Oil during the Ripening of the Olive Fruit of the Chemlal Variety in the Region of Boumerdes (Algeria)

K. Mouas and A. Benakmoum

***Abstract*—** The purpose of this work was to study the variation of the fatty acid composition of olive oil during the ripening of the olive (Olea europaea) fruit of the Chemelal variety in the region of Boumerdes (Algeria).

The olives were picked according to the color of their skin (green, violet and black) in the region of Boumerdes and the extraction was done without any hit ressource whose different mixtures were prepared using a mixture of solvents (Hexane, Acetone and . ethanol) with different proportions. The olive oil extracted after a gas chromatography analysis show that oleic acid is the most abundant at a content of (71.34%), its content increases from 67.94% at the stage of veraison to 71.34% at the stage (mauve) and then decreases to reach the value 69.55% in end of maturity.The content of palmitic, palmitoleic, margaric and stearic acid decreases during ripening whereas the polyunsaturated fatty acid content increases from 9.3% to 10.35%.

***Index Terms*—** Olive, Chemlal, ripening, olive oil, acidity, fatty acids.

I. Introduction

The region of Boumerdes is a region with agricultural vocation; it has an olive orchard with an area of nearly 8,200ha divided largely into a plot of less than 100 olive trees or 700,000 olive trees concentrated in the mountainous areas of the region

Despite the potential that this region can guarantee in terms of typicality and exclusivity of olive production, the oils produced there are decommissioned according to the criteria set by the regulations.

Acidity is the major criterion for classifying oil in different categories (International Olive Council 2011)

Improving the quality of Algerian oil produced with relatively high acidity is a major challenge

According to (Criado, Morello, Motilva and Romero, 2004) the quality of olive oil is influenced by several factors (genetics (cultivars), climatic conditions but also depending on the maturation, because the maturity of olives is a criterion variable whose evolution depends on a set of factors

In addition, maturation advances certain metabolisms resulting in changes in the profile of certain compounds such as fatty acids. (Manual fuente; Sanchez et al 2011).

The aim of this work was to study the variation of the fatty acid composition of olive oil during the maturation of olives of the

Variety Chemlal on the quality of olive oil in the region of Boumerdes.

II. Material And Methods

A. The Sampling

It was realized on a private farm in Chabet el Ameur near Boumerdes from the well irrigated orchards.

3 kg of olives are harvested during ripening from the last week of September to the beginning of February when the olives were picked according to the color of their green, violet and black skin.

After the harvest, the olives were immediately transported to the laboratory, whose characterization of the samples was carried out on: the weight, the length and the width of the olive.

.The maturity index (Ml) was calculated after a visual assessment of the color of the skin on a hundred olives selected at random according to the following formula.

IM = (a X 0) + (b X 1) + (c X 2) + (d X 3) + (e X 4) + (f X 5) + (gx6) + (hx7) / 100

a, b, c are the number of olives in each of the seven color classes that start from dark green to dark black stadium (Hermoso et al 1991).

B. Extraction of the oil

The green and purple olives are washed with tap water and then dried. The extraction of the oil is done cold. For each sample of 250 g of olives of all stages of ripening

, different mixtures were prepared with a mixing of solvents (hexane, acetone and ethanol), the proportions of which represented in table n 01 and 02.

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TABLE I: THE COMPOSITION OF THE MIXTURE FOR GREEN OLIVES

	Hexane %	Acétone%	Ethanol%
Mixture n 01	0	100	0
Mixture n 02	100	0	0
Mixture n 03	50	50	0
Mixture n 04	33.33	33.33	33.33
Mixture n 05	50	00	50

TABLE II: COMPOSITION OF THE MIXTURE FOR PURPEL OLIVES

	Hexane%	Acétone%	Ethanol%
Mixture 01	50	50	0
Mixture 02	33.33	33.33	33.33

For olives at the end of the mature stage, extraction of the oil was carried out using hexane after stirring.

The oil was extracted several times for 30 minutes. The filtrates obtained after each extraction were mixed with the same solvent which will be removed using the rotavapor.

C. Free acidity

It is determined according to the method described in Regulation EEC / 2568/91

An oil sample of 5 g was dissolved in 20 ml of a mixture of diethyl ether-ethanol 95% (V / V). The mixture was titrated by stirring with an ethanolic solution of potassium hydroxide (0.1N) in the presence of phenolphthalein until a persistent pink color ten seconds. A control test was carried out under the same conditions. The acidity is expressed as a percentage of oleic acid which is thus determined:

A% (oleic acid) = (V - V0) * (N * M / 10 * m)

V and V0: volume in milliliter of KOH necessary for the neutralization of the sample and white, respectively. N: normality of the KOH solution (0.1N). M: molar mass of oleic acid which is equal to 282 g / ml. m: mass in grams of the test sample.

D. Chromatographic analysis of fatty acids

The knowledge of the fatty acid composition of olive oil is an important analysis in the determination of its quality, the method of choice is gas chromatography after transformation of the fatty acids into methyl ester (Mariani and Fedeli, 1993).

Transesterification is cold with a methanolic solution

Potassium hydroxide as intermediate phase prior to saponification using the official method (ISO 5509: 2000, point 5 of IUPAC Method 2.301).

With the chromatogramChrompack CP 9002

The carrier gas used is nitrogen and the quantity injected is 0.8ul

The results are expressed in percentage relative to each fatty acid calculated by internal normalization of the area of the chromatographic peak.

III. RESULTS AND INTERPRETATION

A. Characterization of the olive

1. The calibration:

The dimensions of olive fruit harvested at different stages of maturity are measured on a sample of 100 olives which will be weighed later using an analytical balance.

The table n 03 represents the average values of the size and weight.

TABLE III: AVERAGE SIZE OF OLIVES AT DIFFERENT STAGES OF RIPENING

	Green (sep/oct)	Reddish (oct/nov)	Purple (nov/dec)	Black (jan/feb)
Length (cm)	5.5618	5.7008	5.7162	4.8580
Width (cm)	0.7135	0.7408	0.7536	0.6235
Weight (g)	1.4489	1.5165	1.5269	1.3545

The olive is a drupe of shape avoide or ellipsoid of very variable dimensions according to the varieties (Friazluiz et al, 1991).

According to (Mendil and selrai, 2006) the variety Chemelal represents more than 40 percent of the national orchard and characterized by these small fruits (2.5 g)

The results in the table 03 show that the olive of the Chemelal variety has an average caliber of (5.5618 cmin engths and 0.7135cm in width). During the veraison stage, this value has increased to reach (5.7008 cm in engths , 0.7408 cm in width) Figure 01

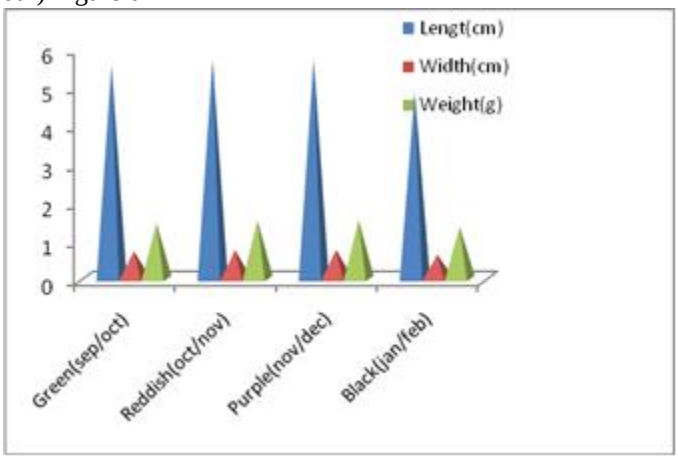

Fig. 1 : calibration of olives

In November especially for the weight that went from (1.5269g). This increase may be due to enzymatic metabolism.

according to (Ajana et al, 1999). the multiplication of the tissues was important and will contribute with the synthesized products to the increase of the weight of the fruits.

From the table we see a clear decrease in weight and size , this observation is in agreement with (Atouati, 1991) for the olives of Marrakesh and (Ajana et al, 1999) who found 3.42 g for the variety picholine from Morocco.

2. The Maturity Indexe:

Maturity index is an important criterion for estimating the most profitable crop (Ajana et al, 1999)

The table 04 shows the results found of maturity indices of olives harvested at different stages of ripening:

TABLE IV: EVOLUTION OF THE MATURITY INDEX

date of sampling	weight	MI
25 /09	1.443	2.12
17/10	1.455	2.58
15/11	1.522	3.25
16/12	1.488	3.79
14/01	1.452	4.25
25/01	1.385	5.12
03/02	1.352	5.45

According to the results of the maturity index, it can be seen that the latter progressively changed over time from 2.58 to 3.25 from October to November, then to 4.25 in mid-January, and continues to evolve to reach the highest value 5.45 in February (Figure 02), however the weight of olives of the variety Chemelal increases slightly as maturation increases.

(Sanchez Casas et al el antari et al 2003 a) believes that progression varies from one variety to another.

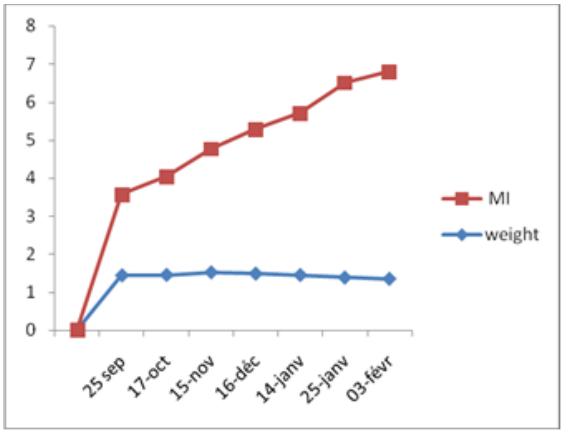

Fig. 2: Evolution of maturity index

B. Characterization of olive oil

1. Solvent effect

The cold extraction of olives olives is done after preparation of the mixing of sovent to evaluate the effect of the solvent on the quality of olive oil.

The results are shown in Table 5 and 6followers.

TABLE V: FATTY ACID COMPOSITION OF GREEN OLIVE OIL OF DIFFERENT MIXTURES

Green olive	acét	hex	Acét /hex	Act/hex /eth
Palmitic	-	17.55	17.59	21.28
Palmitoléic	-	2.15	2.15	3.31
Margarique	-	0.075	0.075	TRACE
Stearic	-	2.075	2.04	2.02
Oleic	-	68.12	67.94	61.19
Linoleic	-	8.40	8.69	10.72
Lenolenic	-	0.62	0.61	0.92
Arachidic	-	0.48	0.43	0.23
Gondoic	-	0.47	0.42	0.28
Behénic	-	0.0004	0.0005	TRACE
SFA	-	19.24	19.19	23.53
MIFA		70.74	70.51	64.78
PIFA	-	9.03	9.30	11.64

TABLE VI: FATTY ACID COMPOSITION OF MAUVE OLIVE OF DIFFERENT MIXTURES

Olives mauves	Acéton/hex	Hex/acét/éth
palmitic	14.92	14.63
Palmitoleic	1.45	1.51
Margaric	0.04	-
Stearic	2.01	1.94
Oleic	71.34	70.36
Linoleic	8.84	9.39
Lenolenic	0.51	0.57
Arachidic	0.4	0.75
Gondoic	0.41	0.47
Behénic	0.0004	trace
SFA	17.61	17.32
MIFA	73.2	72.34
PIFA	9.35	9.96

From the results shown above it is found that acetone is a solvent unable to extract the oil containing in the olive, this may be due to its inability to penetrate the vegetative cell since the olives are pricked and that the Acetone is a solvent used for its property of solubilizing many organic species while hexane was able to extract the oil from the stung olives with a very rich fatty acid composition (68.12% oleic; , stearic and monounsaturated fatty acids, this is necessarily due to its apolar properties which give it a high affinity for fat, the same observation was reported by (Ajana et al; 1999)

In addition, the acetone / hexane / ethanol mixture is the strongest for the extraction of an oil richer in palmitic fatty acid, palmitoleic saturated fatty acid and polyunsaturated Especially for purple olives; the acetone mixture; hexane; ethanol was the most effective at extracting the oil in and chemical composition of fatty acids (figure 03 and 04).

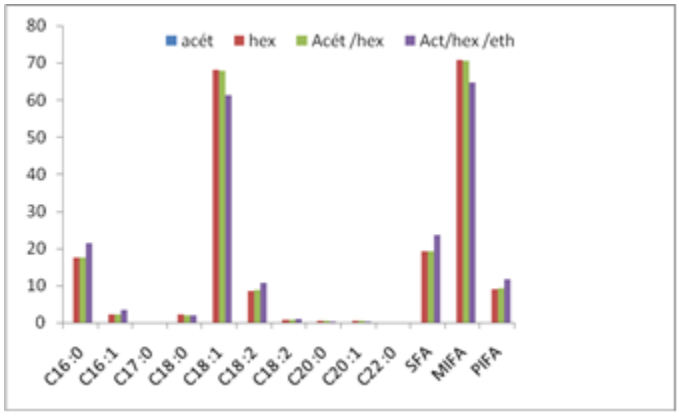

Fig. 03 : solvent effect on green olive oil

SFA: saturate fatty acid. MIFA: monoinsaturé fatty acid.

PSFA: polyinsaturat fatty acid

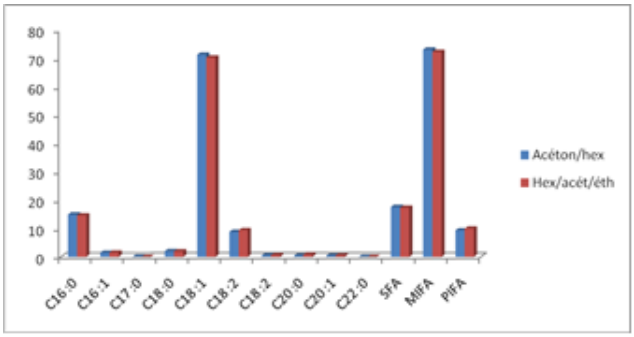

Fig. 04: Solvent effect on purple olive oil

SFA: saturate fatty acid. MIFA: monoinsaturé fatty acid.

PSFA: polyinsaturated fatty acid

2. Acidity:

Acidity is a quality parameter of olive oil it allows us to learn On its state of degradation

The results found of the acidity that are presented in the following table.

TABLE VII: ACIDITY VALUES OF OILS EXTRACTED AT DIFFERENT STAGES OF RIPENING

olive	green	reddish	purple	black
acidity	(sep /oct)	(oct /nov)	nov /dec	(jan /fev)
	0,15	0,152	0,18	0,29

This table shows that the acidity of the olive oil of the variety Chemlal increases during ripening of the olives, contrary to the results found in the work of Sanchez Casas et al. (1999) find that there is no correlation between olive harvest date and acidity

Olive oil extracted from green olives between September and October has an acidity of 0.15 percent. then it increases to 0.18 at the purple stage and at the end 0.29 at the black stage in February figure 05.

This increase is necessarily due to the hydrolysis of triglycerides.

(Michelakis, 1990) considers that the acidid is the result of the hydrolysis of the oil caused by the action of the enzymes released during the ripening of the fruit.

the highest value of the acidity of the olive oil for late black olive does not exceed 0.29 and according to the commercial standard of the International Olive Council (IOC 2003). The Chemlal vareity analyzed is part of the category common virgin olive oil.

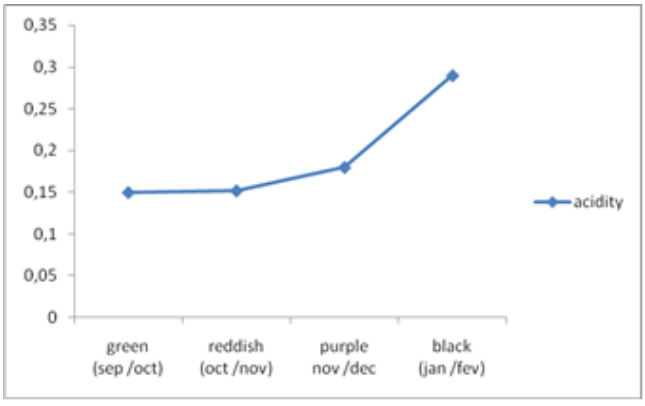

Fig. 05 Evolution of acidity during maturity

3. Composition in fatty acids:

GPC analysis results for the following olive oil samples are shown in the following table 08.

TABLE VIII: VARIATION OF FATTY ACIDS DURING RIPENING

Fatty acid	Green (sep/oct)	Violet (oct/nov)	Black (janv/fev)
C16:0	17.59	14.92	14.91
C16 :1	2.15	1.45	1.44
C17:0	0.075	0.04	0.04
C18:0	2.04	2.02	2.01
C18 :1	67.94	71.34	69.55
C18 :2	8.64	8.84	9.81
C18 :2	0.61	0.63	0.84
C20:0	0.43	0.42	0.42
C20 :1	0.42	0.41	0.45
C22:0	0.0005	0.0004	0.0004
SFA	19.19	17.61	17.38
MIFA	70.51	73.2	71.63
PIFA	9.3	9.35	10.35

The value shown in the table shows that the most abundant oleic acid has a content of 71.34%

The palmitic, palmitoleic, margaric and stearic acid content decreases during maturation (Figure 6).

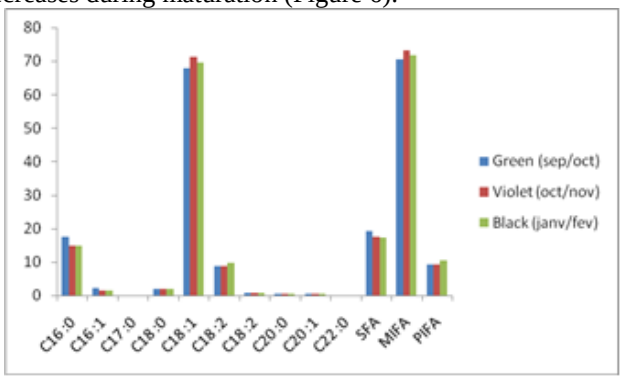

Fig. 06: Variation of fatty acid during maturity

SFA: saturate fatty acid. MIFA: monoinsaturated fatty acid.

PSFA: polyinsaturated fatty acid

These results are similar to those reported by (Tamenjari et al; 2004b) who worked on the chemelal variety, especially in those of (Baccouri et al; 2008) after analysis of virgin olive oil

Monovarietal tunez unlike those reported by (Salvador et al) who worked on a Spanish variety Cornicabra during ripening.

The oleic acid content increases from 67.94% at the stage of veraison to 71.34% at the stage (mauve) and then decreases to reach the value 69.55% at the end of maturity (figure 06), this can be characterized by a lipid synthesis (lipogenesis) during the first months of maturation then to enzymatic degradation at the end of maturity.

(Zarrouk et al; 1996) believes that the change in fatty acid composition is explained by endogenous or exogenous lipolysis.

The results of the fatty acids show us that the ratio oleic / linoleic is high this ratio oleic acid / linoleic is frequently used as parameter of stability (Velasco Dobarganes, 2002).

The polyunsaturated fatty acid content increases during ripening from 9.3% to 10.35% , which is a desirable property for the oxidative stability of the oil.

These results are not similar to those reported by (Manuel Fuente Sanchez et al 2011) and in especially to those of (Angerosa et al 2000) who considers that the low activity of

lipooxygenesis is responsible for the decrease in volatile compounds.

IV. Conclusion

In order to produce good quality olive oil in the Boumerdes region, we have studied the maturation effect on the acidic composition of the olive oil of the Chemelal variety.

We have found that the nature of the solvent has a remarkable effect on the fatty acid composition of the olive oil extracted as to the different roles that each can provide.

This study has made it possible to assess the significant effect of olive fruit ripening on the acidic composition of olive oil. The degree of maturity affects the content of volatile components, especially oleic and palmitic acids. Steroids and saturated fatty acids

This suggests that the optimal period for picking olives of the Chemelal variety to obtain a better acid composition is between the end of October and the end of November, hence the need to change the mentality of some farmers regarding the respect of good harvesting practices.

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