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INFLUENCE OF RIPENING INDEX ON THE YIELD AND QUALITY OF VIRGIN OLIVE OIL IN THE BOUMERDES AREA

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Abstract

Description of the subject: Algeria is considered one of the leading olive growing areas in the world. The maturity index is one of the most important factors contributing to oil quality.
Objectives: This study was conducted to investigate the effect of olive maturity index on the physicochemical properties of oil using the chemlal cultivar in the region of Boumerdes.
Methods: The study was conducted in a commercial chemlal olive planted in the village of chebat el Eumer. Seven samples were collected from 25 September 2018 to 03 February 2019. Each sample was tested for fruit characteristics including moisture maturity index, weight, length and width of fruits. Oil extracted by the mechanical method was tested for acidity, peroxide, UV light extinctions, oil fatty acid composition, pigments, polyphenols and functional analysis by FT-IR.
Results: The oil content ratio increased significantly over the harvest dates, a clear correlation was observed between the colour index and each of the fruit weight ratios. Acidity and peroxide values increased during the harvest dates. K_{232}, K_{272}, ΔK were within the International Olive Oil Council specifications for extra virgin oil. Oleic acid and palmitic acid levels ranged from (64.04-70.63%) and (14.63-21.28%) respectively. Chlorophyll and carotenoids were all (31.07); (1.36) mg/kg respectively for the first ripening index. Total polyphenols show significant differences between harvest dates, the lowest value was observed at the last sampling date (259 mg/kg) and the highest at the date of 25/09/2018 (321 mg/kg).
Conclusion: The results obtained could be useful in determining the optimal for picking olives, which is between the end of September and the end of October for the Chemelal variety.
Keywords: Chemlal variety, Extra virgin olive oil, Polyphenols, Ripening index, Harvest date, fatty acid profile.

INFLUENCE DE L'INDICE DE MATURATION SUR LE RENDEMENT ET LA QUALITÉ DE L'HUILE D'OLIVE VIÈRGE DANS LA RÉGION DE BOUMERDES

Résumé

Description du sujet : L'Algérie est considérée comme l'une des premières régions oléicoles du monde. L'indice de maturité est l'un des facteurs les plus importants contribuant à la qualité de l'huile.
Objectifs : Cette étude a été menée pour étudier l'effet de l'indice de maturité des olives sur les propriétés physico-chimiques de l'huile d'olive de la vareité chemlal dans la région de Boumerdes.
Méthodes : L'étude a été menée sur des olives de la vareité chemelal dans une ferme située à chabet el ameur dans la region de boumerdes. Sept échantillons ont été collectés du 25 septembre 2018 au 03 février 2019. Chaque échantillon a été testé pour les caractéristiques des fruits, notamment l'indice de maturité d'humidité, le poids, la longueur et la largeur des fruits. Les analyses effectuées sur l'huile d'olive extraite à froid portent sur l'acidité, l'indice de peroxyde, les coefficients d'extinctions à la lumière UV, la composition en acides gras de l'huile, les pigments, les polyphénols par CPG et l'analyse fonctionnelle par FT-IR.
Résultats : Le rapport de la teneur en huile a augmenté de manière significative au cours des differnts dates de récolte, une corrélation claire a été observée entre l'indice de couleur et chacun des rapports de poids des fruits. Les valeurs d'acidité et de peroxyde ont augmenté au cours des dates de récolte. K232, K272, ΔK étaient conformes aux spécifications du Conseil oléicole international pour l'huile extra vierge. Les teneurs d'acide oléique et d'acide palmitique variaient respectivement de (64,04-70,63%) à (14,63-21,28%). La chlorophylle et les caroténoïdes étaient de (31,07) ; (1,36) mg/kg respectivement pour le premier indice de maturation. Les polyphénols totaux montrent des différences significatives entre les differents dates de récolte, la valeur la plus faible a été observée à la dernière date d'échantillonnage (259 mg/kg) et la plus élevée à la date du 25/09/2018 (321 mg/kg).
Conclusion : Les résultats obtenus pourraient être utiles pour déterminer la période optimale de cueillette des olives, qui se situe entre fin septembre et fin octobre pour la variété Chemelal.
Mots clés : Variété Chemelal, Huile d'olive extra vierge, Polyphénols, Indice de maturation, Date de récolte, Profil en acides gras.

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INTRODUCTION

The olive (*Olea europaea* L.) is the main fruit cultivated in the Mediterranean basin, and olive oil is a basic diet element in the region [1]. Mediterranean countries are the main producers with 97% of the world olive oil production, estimated at 3.500.000 tonnes for the 2019/2020 campaign. Algeria is the eighth world olive oil producer after Spain, Italy, Greece, Turkey, Morocco, Portugal and Tunisia [2]. Algerian Chemlal is a variety considered as a true local heritage, it yields 14 to 18 litres/quintal [3]. The world olive oil consumption continues to increase, even in countries which have no history of olive growing, and it is generally parallel to the production rate [4]. The significant increase in the demand and olive oil consumption is due to its nutritional value and health benefits, including its antioxidant, anti-atherogenic, anti-inflammatory, anti-tumour, antiviral, anti-cancer and immunomodulating activities [5, 6]. The chemical characteristics and quality of virgin olive oil are influenced by several factors, including genotype, trees age, fruit ripening, production area, soil, climate conditions, agronomic and irrigation practices and the extraction process [7,8]. The quality of virgin olive oil is strongly linked to the fruit ripening. An increase in polyunsaturated fatty acids associated with a loss of total phenols, pigments and oxidative stability, as the olives ripen, has been reported in many studies, [9 ,10 ,11 and 12] have shown reductions in peroxide value, extinction coefficient at 232 and 270 nm, chlorophylls, carotenoids and oleic acid content during ripening. Similarly, Nsir [13] and Piscopo [14] observed a downward trend in quality parameters in the virgin olive oil produced from a more advanced olive ripening stages. Changes in oil content during the fruit ripening process have also been documented in several studies [15,16] In fact, the oil content accumulates in the olives as they ripen [17,18]. In the same trend, Benito [19] and Mena [20] have reported an increase in industrial oil yield during olive ripening. Consequently, the study objectives were to study the olive ripening stage effect on the yield and quality of the (chemlal) Algerian variety virgin olive oil (*Olea europaea* L.) produced in north-central Algeria.

MATERIALS AND METHODS

1. Experimental design and environmental conditions

The present survey was conducted during the 2018/2019 agricultural campaign, on the widely cultivated olive variety "Algerian chemlal" in an experimental olive grove located in the province of (Chabet el Ameur) (Latitude : 36.6333, Longitude: 3.7 36° 37′ 60″ North, 3° 42′ 0″ East) (Boumerdes) town in northern (Algeria) (Fig. 1). The climate is of Mediterranean type, with mild and humid winters and dry, hot summers (Fig. 2). Different olives samples (3 kg for each sample) were taken by hand at random from three olive trees in a private farm representing each treatment at different stages of ripening according to the skin colour degree and pulp. The sampling dates were as follows: 24 September (green skin colour), 6 October (green skin colour with reddish spots), 20 October (red to violet skin colour), 15 November (violet to black skin colour with white flesh), and 27 January (black skin colour with violet flesh).

Figure 1: The location and the coordinates (Latitude: 36.6333, Longitude: 3.7 36° 37′ 60″ North, 3° 42′ 0″ East) of study area (from the Google Earth site).

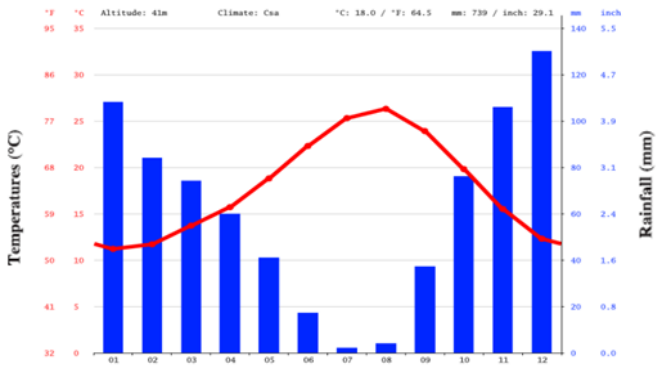

Figure 2: Annual patterns of 2018 year for air temperatures and rainfall in Boumerdes province (northern Algeria). Source Climat-data.org

2. Ripening index determination

The ripeness index (RI), for olives collected at seven maturation stages, was determined according to the method developed at the Agronomic Station of Jaén (Spain) [21]. It is based on a scoring system for each stage of colouring of the skin and flesh (Fig. 3). The RI was obtained on 100 randomly olive fruits in each sample freshly picked, by applying the following formula: $RI = \frac{(\eta 0*0)+(\eta 1*1)+(\eta 2*2)+\cdots+(\eta 7*7)}{100}$. Where n_1, n_2, n_3, n_4, n_5, n_6, n_7, represent the number of olive fruits that belong to the following eight categories:

0: Olive fruits number with green epidermis.
1: Olive fruits number with yellow or greenish-yellow epidermis.
2: Olive fruits number with yellow skin with dots.
3: Olive fruits number with red or light purple skin.
4: Olive fruits number with black skin but with stone green.
5: Olive fruits number with black skin but with purple stone up to half.
6: Olive fruits number with black skin and almost stone completely pink.
7: Olive fruits number with black skin and totally dark stone.

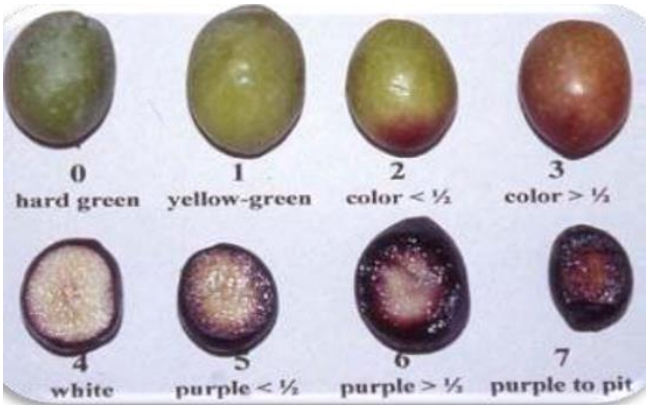

Figure 3: Variation in the colouring of the olive epicarp during ripening. [65]

2.1. Olive average weight

The olive average weight is used to evaluate the fruit size. A sample of one hundred fruits is taken at random and then weighed by an analytical balance type: PA214C Ohaus™ 8729258208 corp.pin Brook Nj USA [22].

2.2. Color analyses

The olive fruits color was measured by Minolta Chroma meter Cr-10 color meter (Minolta Co., osaka, Japan). It was calibrated against a standard calibration plate of a white surface and set to CIE Standard Illuminant C. The L*, a*, b* values are the averages of three readings. The color brightness coordinate L* measures the whiteness value of a color and ranges from black at 0 to white at 100.The chromaticity coordinate a* measures red when positive and green when negative and the chromaticity coordinate b* measures yellow when positive and blue when negative [23].

2.3. Fruit moisture content

Ten olives from each ripening period were placed in a petri dish and weighed before being placed in an oven at 105°C for 48 hours until they reached a constant mass. The water content was determined in triplicates from the difference between fresh and dry mass and expressed as a percentage [24].

3. Cold oil extraction

The healthy olive fruits hand-picked at different ripening stages from trees were immediately transported to the laboratory. Olive oil extraction was performed at the Laboratory of food technology of the M'hamed Bougara university Faculty of Technology (Boumerdes) using a lab-scale instrument reproducing industrial conditions for oil extraction, olives were crushed with a hammer crusher, the resulting paste was slowly mixed at room temperatures for 30 min, and the oil was separated by centrifugation (3000 rpm over 5 min) without addition of warm water.

The obtained oil was filtered, transferred into amber glass bottles without headspace, and stored in the dark at 4 °C until analyses. The industrial oil yield (extracted oil), given in percentage of fresh olive paste weight (W) and considering the olive oil density (D) at ambient temperature of 0.915 g.mL^{-1} , was determined using the formula [20]. Extracted oil (%) = $\frac{V*D}{W} * 100$. Where V is the olive oil volume obtained (mL).

4. Olive oil analysis

4.1. Quality indices

Determination of free fatty acids, peroxide value, and specific wavelength absorbance at 232 nm and 270 nm (K_{232} and K_{270}) were carried out, following the analytical methods described in Regulations EEC/2568/91 and later modifications of the Commission of the European Union [22]. Free fatty acids, expressed as% of oleic acid, was determined by titration of a mixture of oil sample (20 g) dissolved in ethanol (50 mL) with ethanolic solution of potassium hydroxide (0.1 N). Phenolphthalein was used as indicator. Peroxide value, expressed in milliequivalents of active oxygen per kilogram of oil (meq O_2/kg), was measured in the following procedure: olive oil sample (5 g) was dissolved in a solution of chloroform-acetic acid (30 mL) then the mixture was left to react with a solution of potassium iodide in darkness. The liberated iodine by the peroxides was titrated with standardized sodium thiosulphate solution using starch as indicator. K_{232} and K_{270} were calculated from absorption at 232 and 270 nm, respectively, with a UV spectrophotometer (UV-1800 SHIMADZU), using a 1% solution of olive oil in cyclohexane (1 g/100 mL) and a path length of 1 cm.

4.2. Pigments

The pigment contents (mg/kg of oil) were determined colorimetrically using UV-1800 SHIMADZU, following the method described by Minguez-Mosquera [25]. A sample of 7.5 g oil was dissolved in 25 mL of cyclohexane. The absorbance of this solution was read at 670 and 470 nm for chlorophylls and for carotenoids, respectively. The values of the specific extinction coefficients used were 613 for pheophytin as major component in the chlorophyll fraction, and 2000 for lutein as major component in the carotenoid fraction.

Thusly, pigment contents were calculated using the following formulas : $Chlorophylls\left(\frac{mg}{kg}\right) = \frac{A_{670}*10^6}{613*100*L'}$; $Carotenoids\left(\frac{mg}{kg}\right) = \frac{A_{470}*10^6}{2000*100*L'}$.
Where A is the absorbance.
L'is the spectrophotometer cell thickness (1 Cm).

4.3. Total phenols

Total phenols were isolated according to the method described by Zunin [26]. Olive oil samples (10 g) dissolved in n-hexane (10 mL) were extracted three times with aqueous methanol (60/40, v/v, 10 mL). The concentration of total phenols was determined spectrophotometrically UV-1800 SHIMADZUfollowing the method of Folin [27]. The latter reagent was added to a suitable dilution of the extract, and the absorbance was measured at 750 nm using as standard the caffeic acid (SigmaAldrich, St. Louis, MO, USA). Values for total phenols content are given as mg caffeic acid/kg oil.

4.4. Specifique phenols

Specifiques phenols (as mg kg-1) extraction for HPLC analysis was carried out according to Caponio [28] and Makhlouf [29] Approximately 5 g (± 0.001) of EVOO was weighted in a 50 mL plastic falcon and 1 mL of hexane and 2 mL of MeOH/water mixture (70 :30 v/v) were added together with 250 mL of an internal standard solution of gallic acid (100 mg L^{-1}in MeOH/water 70 :30, v/v). After mixing, samples were centrifuged for 10 min at 4°C at 3940 × g (SL 16R Centrifuge, Thermo Fisher Scientific Inc., Waltham, MA, USA). The methanolic fraction was collected and centrifuged for 5 min at 4°C at 8870 × g, then filtered with 0.45 µm nylon filters (VWR International, Center Valley, PA, USA). The chromatographic system and conditions were those reported in [30]. Phenolic compound were identified by comparing the retention times with those of the reference standards, or with literature data where no standards were available.

4.5. Infrared analysis (FTIR)

FTIR spectra were obtained using a Alpha Bruker FTIR Spectrophotometer equipped with an attenuated total reflectance accessory (ATR single-reflexion, Diamond, incident angle 45◦C), DTGS detector, Globar (MIR) source and KBr Germanium separator, with a resolution of 0,1-0.5 cm^{-1}at 98 scans.

Spectra were scanned in the absorbance mode from 4000-375 $Cm^{-1}$and the data are handled with OPUS logiciel [31].

4.6. Fatty acids analysis by GC-MS

The analyses of fatty acids were determined using oils Methylation with borontrifluoride methanol method [32]. About150 mg of oil, 6mL of KOH in 0.50 $molL^{-1}$ methanol was added and agitated with heat for 5 minute. After that 5.0 mL BF_3(14%) in methanol was added with agitated under heat for 3minute. Next 3.0 mL of isooctane and about15.0 mL of saturated sodium chloride were added and strongly agitated for1 5seconds.After phase's separation, the upper layer, containing the methyl esters offatty acids was collected. The analyses by gas chromatography coupled with mass spectroscopy of fatty acid methyl esters were carried out on a Hewlett Packard Agilent 6890 plus gas chromatograph equipped with a mass detector (Hewlett Packard Agilent 5973). The column is Type: HP-5MS, 30 metres long and 0.25 mm in diameter with a film thickness of 0.25 µm, its stationary phase is 5% Phenyl 95% dimethylpolysiloxane. The carrier gas is Helium with a purity: N6; GV flow rate: 1 ml/min). The column temperature programme is Oven temperature: 70°C for 5 min, 10°C/min up to 130°C, isothermal for 2 min, 3°C/min up to 220°C, isothermal for 4 min, 10°C/min up to 280°C, isothermal for 7 min. The samples were injected in Splitless mode. The apparatus itself performed the recording and integration. The gas chromatographic peaks were identified as the corresponding fatty acid methyl esters by checking the order of elution on the column and compared the retention times with those of the pure standards.

5. Statistical analyses.

Olive and oil analyses data were submitted to one-way analysis of variance ANOVA with harvesting time as group variable. The software XLSTAT 2014 was used to process data. Means, when required, were separated according to Tukey's test, significance level $p \leq 0.05$.

RESULTS

1. The effect of harvest date on fruit characteristics

1.1. Ripening index

The olive ripeness index values increase continuously and significantly. Indeed, the values recorded are between 2.12 and 5.45. This increase is recorded from the month of September until the beginning of February.

1.2. Moisture content

According to Table 1, the moisture content of Chemlal variety fruit changes irregularly during ripening. In fact, this rate remains relatively constant at the beginning, 56.44 and 57.13% respectively, then increases a little bit to 59.55%, then decreases rapidly, and finally this decrease becomes less important 54.66%.

1.3. Average fruit weight, length and width

The continuous evolution of the fruit weight according to the seven harvest periods is illustrated in table 1. The fruit weight shows values ranging from 1.44g (RI 2.12) to 1.95g (RI 4.25). The average values for fruit length vary between 1.09 mm and 1.13 mm for this variety. The results of average weight, length and width of the olive fruit during the seven harvesting dates was 1.73 g, 2.12 cm, 1.11 cm, respectively.

1.4. Color analysis

Color values (L, a and b values) were measured during storage. The maximum for L is 100, which would be a perfect reflecting diffuser. The minimum measuring for L would be zero, which would be black. The a and b have no specific numerical limits. Positive a is red and negative a is green. Positive b is yellow and negative b is blue. Analysis of color (L, a and b values) showed that the color of olive oil samples changed significantly during storage (Tab. 1). The lowest L value (lightness) was seen in the eighth month. The L values was increased to 34.15 after 9 months and then decreased lightly. Fluctuations were observed in a (redness) and b (yellowness) values during storage. The highest b value was obtained for the tenth month. After this month there was a decreasing trend in b value. The highest a value also observed in ninth and twelfth months.

1.5. Oil content

In this study, as shown in Table 1, the oil olives content (% ms) increases from 29% (RI 2.12) to 48% (RI 3.79), so a rate of 19%, and then decreases to a minimum value of 33% at the last harvest date (7) (RI 5.45). However, statistical analysis revealed a significant difference ($p < 0.05$).

2. The effect of harvest dates on the olive oil chemical characteristics related to oil quality

With reference to the COI (2011) standard on the virgin olive oil quality,

the quality indices (acidity, peroxide value, Abs at 232 and 270 nm) allow virgin olive oil to be classified into different oil categories. These standards are applied in the case of trade. Table 2 shows the results of the quality indices of the oil extracted from olives harvested at different dates, corresponding to the different stages of maturity. These results reveal in fact that all the oil samples analysed, regardless of the dates of olive harvesting, belong to the category of extra virgin olive oil.

2.1. Acidity level

Referring to the results of Table 2, the acidity level of virgin olive oil tends to increase slightly during maturation. In fact, the acidity rate increases from 0.3% in the first harvest (RI 2.12) to 0.4% in the last harvest (RI 5.45). However, the analysis of variance did not reveal a slight significant difference.

2.2. Peroxide value

The evolution of this index shows a slight increase and then a decrease to reach at the end a maximum value of12.39 meq g O_2/kg at (RI 5.45). Statistical analysis revealed a significant difference (p<0.05).

2.3. Absorbance in ultra violet

K232 values follow the same evolution as the peroxide index (R^2 = + 0.71). Indeed, the lowest value (2.04) was recorded on the third harvest date (IR 3.25) and the highest value (2.72) was obtained on the fourth harvest date (IR 3.79). K270 is another quality parameter evaluated, and considered significant for secondary oxidation. The results of this parameter do not vary significantly between different harvest dates.

2.4. Pigment content

From the first stage of olive ripening corresponding, in this study, the chlorophyll content shows a significant (p<0.001) decrease, from 31.07 to 11.02 mg/kg. The carotenoid content of virgin olive oil obtained from olive at different stages of maturity does not vary significantly. In fact, their contents vary from 1.23 mg/kg (IR 3.79) to 1.55 mg/kg (IR 4.25).

2.5. Total phenolic compounds

The results show that the Chemelal variety presents considerable levels of total polyphenols. The total phenolic compound decrease significantly during the ripening of the olive (p < 0.05). Indeed, the highest content (321.43 mg/kg) is obtained at the first harvest date which corresponds to IR of 2.12, this value decreases to 259 mg/kg obtained at a late stage of ripening corresponding to a (IR 5.45).

Table 1: Olive fruit characterisation according to the ripening index

	Chemlal variety						
Date	25-09-2018	17-10-2018	15-11-2018	16-12-2018	14-01-2019	25-01-2019	03-02-2019
Repening index	1(RI=2.12)	2 (RI=2.58)	3 (RI=3.25)	4 (RI=3.79)	5 (RI=4.25)	6 (RI=5.12)	7 (RI=5.45)
Dry matter (%)	56.44± 3.22 $^{(a,b)}$	57.13±3.65 $^{(a,b)}$	59.55± 3.77$^{(a)}$	54.66±2.98 $^{(a,b)}$	52.14± 2.02$^{(b)}$	51.53± 2.09$^{(b)}$	54.66±2.11$^{(b)}$
Fruit weight (g)	1.44±0.02$^{(e)}$	1.55±0.03$^{(d)}$	1.72±0.04$^{(c)}$	1.88±0.03 $^{(a,b)}$	1.95±0.02$^{(a)}$	1.83±0.02$^{(b)}$	1.75±0.02$^{(c)}$
Fruit width (Cm)	1.09±0.01$^{(a)}$	1.09±0.01$^{(a)}$	1.11±0.02$^{(a)}$	1.13±0.01$^{(a)}$	1.12±0.02$^{(a)}$	1.11±0.02$^{(a)}$	1.09±0.01$^{(a)}$
Fruit length (Cm)	2.02± 0.01$^{(d)}$	2.06±0.02$^{(c,d)}$	2.09±0.02 $^{(b,c)}$	2.11±0.02$^{(b)}$	2.21±0.02$^{(a)}$	2.19±0.01$^{(a)}$	2.18±0.02$^{(a)}$
L*	48.02±1.82$^{(a)}$	39.01±1.6$^{(b)}$	34.80±1.61 $^{(b,c)}$	32.71±1.41$^{(c,d)}$	34.41±1.93 $^{(b,c)}$	29.01±1.23$^{(c,d)}$	27.61±1.91$^{(d)}$
a*	-11.01±1.44$^{(b)}$	8.04±1.01$^{(a)}$	10.08±1.31$^{(a)}$	9.02±1.92$^{(a)}$	9.61±1.62$^{(a)}$	8.02±1.13$^{(a)}$	10.05±1.51 $^{(a,)}$
b*	23.53±1.75$^{(a)}$	7.10±1.09$^{(b)}$	7.09±0.91$^{(b)}$	3.51±0.95$^{(c)}$	7.42±0.96$^{(b)}$	2.14±0.44$^{(c)}$	4.71±0.32 $^{(b,c)}$
Total oil extraction (%)	29±2$^{(d)}$	32±3$^{(c,d)}$	37±4$^{(b,c)}$	48±5$^{(a)}$	43±4$^{(a,b)}$	40±3$^{(a,b,c)}$	33±2$^{(c,d)}$

Table 2: Olive oil characterisation according to the ripening index

	Chemlal variety						
Date	25-09-2018	17-10-2018	15-11-2018	16-12-2018	14-01-2019	25-01-2019	03-02-2019
Repening index	1(RI=2.12)	2 (RI=2.58)	3 (RI=3.25)	4 (RI=3.79)	5 (RI=4.25)	6 (RI=5.12)	7 (RI=5.45)
Free fatty acid (%)	0.39±0.025$^{(a)}$	0.47±0.019$^{(a)}$	0.33±0.05$^{(b)}$	0.20±0.03$^{(c)}$	0.40±0.02 $^{(a,b)}$	0.37±0.03$^{(b)}$	0.40±0.015 $^{(a,b)}$
Peroxide value (meqO_2/kg DM)	3.18±0.12$^{(c)}$	3.60±0.51$^{(c)}$	1.43±0.02$^{(d)}$	2.87±0.19$^{(c)}$	3.81±0.11$^{(c)}$	6.87±0.37$^{(b)}$	12.39±0.84$^{(a)}$
K232	2.19±0.02$^{(d)}$	2.54±0.042$^{(c)}$	2.04±0.052$^{(e)}$	2.72±0.02$^{(b)}$	2.20±0.04$^{(d)}$	8.07±0.03$^{(a)}$	2.60±0.01$^{(c)}$
K270	0.17±0.01$^{(d)}$	0.33±0.03 $^{(a,b)}$	0.21±0.05$^{(c,d)}$	0.31±0.04 $^{(a,b,c)}$	0.38±0.06$^{(a)}$	0.27±0.04 $^{(b,c,d)}$	0.2±0.01$^{(d)}$
Carotenoids (mg/kg)	1.36±0.13$^{(a)}$	1.35±0.14$^{(a)}$	1.31±0.15$^{(a)}$	1.23±0.16$^{(a)}$	1.55±0.18$^{(a)}$	1.42±0.21$^{(a)}$	1.39±0.17$^{(a)}$
Chlorophylls (mg/kg)	31.07±2.44$^{(a)}$	29.08±2.22$^{(a)}$	23.01±2.11$^{(b)}$	21.91±0.91$^{(b)}$	16.13±1.12$^{(c)}$	13.06±0.98$^{(c,d)}$	11.02±0.87$^{(d)}$
Total phenolics (mg gallic acid/ kg fresh oil)	321.34±10.33$^{(a)}$	310.21±9.38$^{(a)}$	298.51±11.61 $^{(a,b)}$	279.13±9.53$^{(b)}$	221.09±9.52$^{(c)}$	288.15±8.45$^{(d)}$	259.11±6.89$^{(e)}$

2.6. Individual phenolic compound

The results of the HPLC analysis reveal a qualitative composition very rich in individual phenolic compounds and similar at different maturity stages, notably hydroxytyrosol, tyrosol and euloropéine, phenolic acids (salicylic and vanillic acid) and flavonoids (kaempferol, epicatechin, quercitin, myricetin). Our olive oil samples show a clear difference in the contents of individual phenolic compounds at different ripening stages.

The highest levels of phenolic compounds are recorded for olives in the green stage between the months of September and October for all phenolic compounds, especially phenolic acids (salicylic and vanillic acid (50 and 70 mg/kg respectively) and flavonoids, as shown in Figure 4.

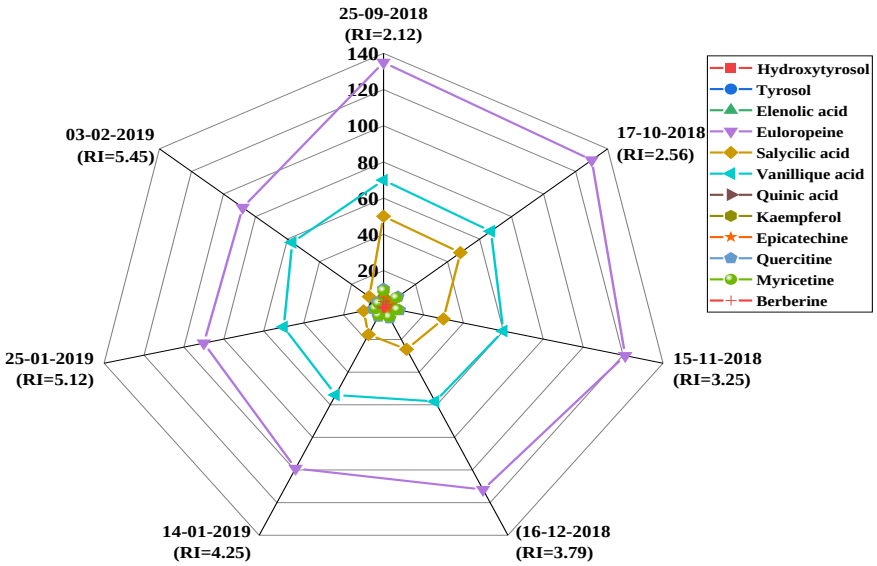

Figure 4: Individual polyphenols variation during maturation.

2.7. Infrared analysis IF-TR

The characteristic FT-IR absorption spectra of the different stages of olive ripeness and their corresponding band assignments for specific functional groups are shown in Figure 5.

Visual inspection of the spectra showed a broad similarity in their spectral profiles throughout the mid-region IR (4000-1000 Cm^{-1}) (Fig 5).

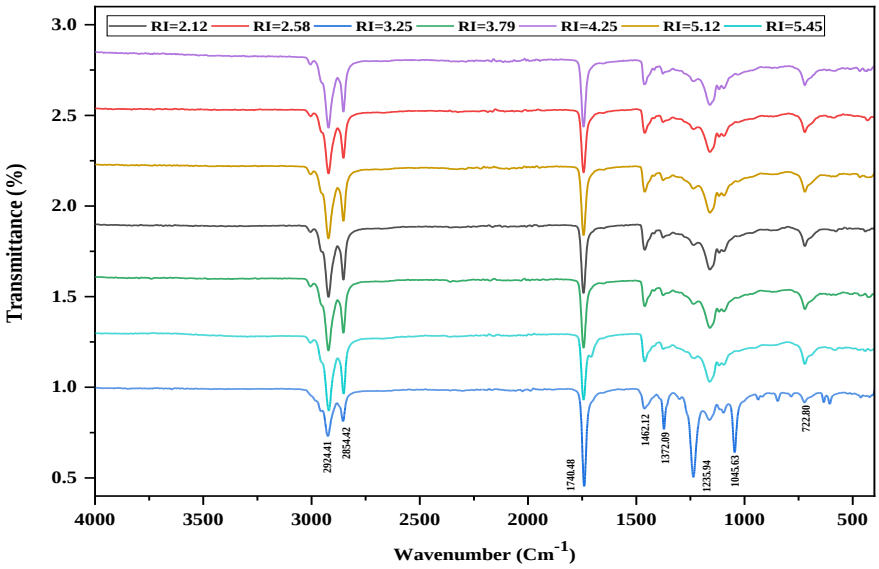

Figure 5: Olive oil FTIR spectra at different maturity stages.

2.8. Fatty acid composition

The results obtained from the gas chromatography are presented in the table below. The results obtained for the seven stages of maturity show that the fatty acid composition of the olive oils analysed meets the standards set by the International Olive Oil Council (OCL, 2009). This acidic composition is variable. Indeed, the percentages of oleic acid (C18:1) vary between 61.19% for IR 3.79 and 70.63 for IR 3.25, while the percentages of palmitic acid (C16:1) vary between 17.72% for IR 4.25 and 14.63% for IR 5.12.

These two fatty acids are therefore the predominant ones, followed by linoleic (C18:2) and stearic (C18:0) acids. The minor fatty acids, whose percentages obtained for the samples studied hardly exceed 4%, are formed by palmitoleic, stearic, linolenic and arachidic acid, while the fatty acids present in trace amounts, whose percentages are lower than 0.2%, are represented by behenic acid. It should also be noted that the fatty acid composition obtained reveals a predominance of monounsaturated fatty acids. The percentage of monoinsaturated fatty acids (MIFA) varies slightly, depending on the samples studied. It varies between 64.78% for IR 3.79 and 73.72% for IR 3.25. Similarly, the percentage of saturated fatty acids varies between 17.80% for IR5.12 and 24.03% for IR3.79. In this study, as shown in Table 1, the oil olives content (% ms) increases from 29% (RI 2.12) to 48% (RI 3.79), so a rate of 19%, and then decreases to a minimum value of 33% at the last harvest date (7) (RI 5.45). However, statistical analysis revealed a significant difference ($p < 0.05$).

Table 3: Fatty acids variation during ripening

Ripening index	2.12	2.58	3.25	3.79	4.25	5.12	5.45
Palmitic C16:0	17.57	17.55	16.09	21.28	17.72	14.63	16.68
Palmitoléic C16:1	2.15	2.15	2.91	3.31	2.44	1.51	2.11
Margarique C17:0	0.08	0.075	0.05	-	0.13	0.42	0.17
Stearic C18:0	2.06	2.075	2.30	2.02	2.14	1.94	2.94
Oleic C18:1	68.03	68.12	70.63	61.19	70.27	70.36	64.06
Linoleic C18:2	8.55	8.40	7.30	10.72	6.78	9.39	10.55
Lenolenic C18:3	0.62	0.62	0.14	0.92	0.32	0.57	1.99
Arachidic C20:0	0.46	0.48	0.39	0.23	0.4	0.75	1.01
Gondoic C20:1	0.45	0.47	0.18	0.28	0.11	0.47	0.45
Behénic C22:0	0.03	0.05	0.03	0.05	0.05	0.06	0.07
SFA	20.20	20.23	19.86	24.03	20.44	17.80	20,87
MIFA	70.63	70.74	73.72	64.78	72.82	72.34	66.62
PIFA	9.17	9.03	7.44	11.64	7.1	9.96	12.54

***SFA**: saturate fatty acid, **MIFA**: monoinsaturé fatty acid, **PSFA**: polyinsaturat fatty acid.*

DISCUSSION

1. The effect of harvest date on fruit characteristics

1.1. Ripening index

The intensity of the chemelal olives variety color varies, during the ripening process, from green to black, and can be expressed by the ripeness index. This latter is measured for the choice of the right date for olives harvesting, which is of great agronomic and economic importance since it determines, on the one hand, the yield and quality of the oil produced and, the production of the next season Rotondi [10] and Conde [33].

Aajana [34], estimated to have no change in this index before the month of September to expect a maximum value of 4.55 at the end of January during a study on the the Moroccan Picholine variety. The increase in this index during ripening can be explained by the progressive degradation of chlorophyll and the accumulation of anthocyanins, [35, 15]

The speed of theindex change of maturity can also be influenced by many other factors, such as the variety of olive tree, the position of the olives on the tree, and environmental factors [36, 37].

1.2. Moisture content

The olives water content is related to their biological development, but also to environmental factors, such as irrigation, rainfall and temperature [36]. Several authors have recorded a decrease in the water content of olives during ripening in particular (Gutierrez-Rosales [38]; Dag [37]). According to Dais and Hatzakis, [39]), the olives moisture content is a very important technological criterion. Indeed, a high water content in the olive can reduce the efficiency of oil extraction, which may result in a loss of flavour and a decrease in the polyphenol content of the oil. Thus, monitoring the moisture content, maturity index and oil content during the ripening of the olives allows olive oil producers to determine the right time to harvest the olives.

1.3. Average fruit weight, length and width

This Work on the olive weight factor has shown that the weight of the olives increases according to the date of harvest. A similar study at the same harvest period shows that the weight of the fruit is increasing [40].

This difference is due to several agronomic and technical factors: ploughing, irrigation, fertilization [41]. ANOVA statistical analysis followed by Tukey's test reveals a significant difference. The results obtained on the fruit length parameter showed that the length of the olives increases to reach a maximum length towards the middle of December followed by a slight decrease towards the end of the harvest.This difference is due to several agronomic and technical factors [42] the statistical analysis does not reveal any significant difference. The average values for the fruit width vary almost as much as those for the lengths. The results of average weight, length and width of the olive fruit during the seven harvesting dates, agrees with the results by Omar [43] who studied fruit characteristics of NB CV in Palestine.

1.4. Color analysis

Although the color is not regarded as an important quality feature for olive oil, it has a great influence on consumer acceptance. The color of olive fruit is depended on olive maturity and process conditions. The change of color during storage has been attributed to decomposition of color pigments such as chlorophylls, pheophytins, xanthophylls and carotenes [44].

1.5. Oil content

The increase in oil content can be explained by the continuedoil biosynthesis during maturation. Indeed, several authors have reported the oil continuation biosynthesis during maturation [36, 45] In addition, the decrease in oil content from the date of 16-12-2018, has also been observed in other olive varieties. Indeed, in the case of the Azeradj and Chetoui varieties, the decline in oil content was observed from RI 3.3 and RI 4.5 respectively [12, 46]. This decline can be explained by the degradation of the oil at this stage of maturity [12, 47]. Finally, it can be concluded that the olive harvest at the end of December, corresponding to an IR of 3.79, allows for the highest oil yield (48%). The study of the Moroccan variety by Ajana [34] showed that the maximum oil content was recorded in the months of December and January. The decreasing in moisture content and increasing oil content was in agreement with previous studies, [48] and was contrary to the findings of by Bengana [49] that appeared there is no significant differences on the oil content of the Chemlal olive oil during the harvest dates.

2. The effect of harvest dates on the olive oil chemical characteristics related to oil quality

2.1. Acidity level

Acidity is a fundamental indicator of the quality of olive oil, and provides information on the hydrolysis of triglycerides [50] the results found in particular the increase in acidity during maturation corroborate those found by several authors [10, 33, 51, 52]. According to Martinez-Suarez [53], the relatively high acidity levels recorded in the late stages of maturity are due to the action of endogenous lipases on triglycerides by releasing free fatty acids.

2.2. Peroxide value

The peroxide value measures total hydro-peroxides, which are primary oxidation products of unsaturated fatty acids [54] the values of the this index found at the different stages of maturation are lower compared to the other results, in particular Vekiari [55] reveal that Peroxide values were rather high and increased with harvesting period for the Throumbolia variety. The evolution of this oil quality parameter was correlated with the lipoxygenase activity in the olive [56].

2.3. Absorbance in ultra violet

K232 values mean that the olive oil extracted from late harvested olives is slightly oxidised compared to that extracted from olives harvested in the early stages of maturity. The results of K270 do not vary significantly between different harvest dates. which means that secondary oxidation did not take place during the ripening of the olives. The results of K232 and K270 recorded in this study corroborate those found in K232 and K270. by [51, 52] In addition, other authors [15, 12] have obtained different results from the evolution of these two criteria, K232 and K270, during the ripening of the olives, and which would probably be due, according to Garcia [57], to the different metabolic pathways of fatty acid oxidation inthe different types of cultivars.

2.4. Pigment content

Chlorophylls and carotenoids are the main pigments in virgin olive oil and are responsible for its characteristic colour. The colour of the oil is one of the factors influencing the consumer choice, and is therefore considered a quality parameter [58]. Everal authors have confirmed the decrease of the chlorophylls concentration in particular ajana [34].

The change in the colour of the olives during ripening is due to a decrease in the chlorophyll content and the accumulation of other pigments, mainly anthocyanins [59] the progressive disappearance of the green colour during olive ripening is due to the decrease in the photosynthetic activity of the chloroplasts and the enzymatic degradation of chlorophyll by chlorphyllases. Moreover, during oil conservation, chlorophyll, which is a photosensitizer, acts as an antioxidant in the dark [60] and a prooxidant in the light [61] therefore the variations in the concentration of this pigment in the oil, associated with inappropriate storage conditions, can negatively influence the shelf life of extra virgin olive oil [59]. Carotenoids are very effective inhibitors of photo-oxidation induced by chlorophyll pigments [62]. The results of carotenoid content of virgin olive oil obtained from olives at different stages of does not agree with those reported by many authors, who point out a decrease in carotenoid content during olive ripening [63] The stability of the carotenoid content, obtained in this study, could be related to varietal character.

2.5. Total phenolic compounds

The high contents of total polyphenols found for the chemlal variet do not agree with those found by Tamendjari [64] who used the same variety, which is necessarily due to the extraction system because we did not use hot water, which could keep the maximum of polyphenols, bound to the polysaccharides of the cell walls. The amounts of total phenols are strongly dependent on the variety [65] In addition, and in addition to their antioxidant activity, phenolic compounds contribute to the bitter and pungent taste, positive attributes, of the extra virgin olive oil [66, 67]. Thus, the loss observed of phenolic compounds during olive ripening confirme the results found by (Gutierrez-Rosales [68], Dag˘delen [69]. Thus, that could negatively affect the sensory quality and oxidative stability of extra virgin olive oil.

2.6. Individual phenolic compound

According to the HPLC analyzes the values found are not in agreement with the results found by (Brenes *et al.*,[70] who noted that the contents of hydroxytyrosol, tyrosol and luteolin in olive oil increase with the progress of fruit ripening, however they are in agreement with those found by (laribi [67]Several authors have explained the decrease in polyphenols during fruit ripening [21] considers that it is probably due to the decrease in PAL activity during the olive ripening process, while (Gandual Roja [71]) finds that it is strongly due to the activity of peroxidase which catalyses the oxidation of these phenolic compounds by the hydrogen peroxide-dichlorophenol system. (Gandul-Rojas and Minguez- Mosquera [72] had explained this decrease by the oxidation of the phenolic compounds by the peroxidase which strongly affects them. However, for other authors such as laribi [67] who used other varieties (Blanquette and Takesrit), there is no change in the concentration of hydroxytyrosol between the spotted and purple stages. Then there is a decrease from the violet to the black stage. This is strongly due to differences in the enzyme systems of each vareity.

2.7. Infrared analysis IF-TR

Similar to those reported by Abdul Rohman [73]. The main absorbance signals included the 3010 Cm^{-1} band associated with the =C-H stretching of cis olefins, the 2900-2800 Cm^{-1} range associated with the symmetrical and asymmetrical stretching of C-H (CH_2 and CH_3), the 1741 Cm^{-1} centred band associated with the stretching vibration of the (-C=O) triglycerides, and the 1461 Cm^{-1} band associated with the C-H bending (shear) vibration of the CH_2 group. The 1376 Cm^{-1} band corresponds to the C-H (symmetrical) bending vibration of the CH_3 group, and the shoulder band centred at 1417 Cm^{-1} due to the rocking vibrations of the C-H bonds of the cis-disubstituted olefins. Finally, the fingerprint region from 1200 to 1000 Cm^{-1} represented the unique stretching and bending vibrations of the -C-O and -CH_2- vibration modes.

2.8. Fatty acid composition

The presence of the polyunsaturated fatty acid linoleic acid (C18:2) with a high percentage compared to other unsaturated fatty acids can be explained by the presence of an enzyme, Oleate desaturase, which transforms oleic acid (C18:1) into linoleic acid (C18:2) during the ripening of the fruit [31] The percentages of oleic acid in the studied olive oils are similar to the values found by Abu-Reidah [74] who found values ranging from 67.24 to 72.27% for Palestinian oils [75]. However, they are somewhat higher than the values reported by Issaoui [76] for Tunisian oils (54.6 to 66.8 %). The ratio of unsaturated fatty acids to saturated fatty acids (UFA/SFA) also shows a fluctuation depending on the samples studied. This ratio varies between 3.18 for IR 3.79 and 4.62 for IR 5.12.

This ratio is higher, which gives olive oil a greater stability to auto-oxidation and an important nutritional value [77, 78]. The increase in oil content can be explained by the continuedoil biosynthesis during maturation. Indeed, several authors have reported the oil continuation biosynthesis during maturation [36, 45] In addition, the decrease in oil content from the date of 16-12-2018 corresponding to RI 3.79, has also been observed in other olive varieties. Indeed, in the case of the Azeradj and Chetoui varieties, the decline in oil content was observed from RI 3.3 and RI 4.5 respectively [12, 46]. This decline can be explained by the degradation of the oil at this stage of maturity [12, 47]. Finally, it can be concluded that the olive harvest at the end of December, corresponding to an IR of 3.79, allows for the highest oil yield (48%). The study of the Moroccan variety by Ajana [34] showed that the maximum oil content was recorded in the months of December and January. The decreasing in moisture content and increasing oil content was in agreement with previous studies, [48] and was contrary to the findings of by Bengana [49] that appeared there is no significant differences on the oil content of the Chemlal olive oil during the harvest dates.

CONCLUSION

The present research work provides information on the maturation patterns of the northern Algerian chemlal variety, grown in location in Algeria (Chabat el ameur), and how it is influenced by the maturity degree during seasonal cultivation. According to the results, progress in maturity was accompanied by a slight increase in oil content acidity and peroxide value, while water content decreased. As the ripening process progressed, a series of changes occurred, including changes in the fatty acid profile (oleic and palmitic acids decreased as ripening progressed, while linoleic acid increased, the MUFA/PUFA ratio also decreased during ripening). A significant decrease was observed in the phenol content and bitterness intensity. It has been shown that the growing season is also an essential factor in determining the chemical composition of olive oil. Given the impact that polyphenols have in inhibiting the different mechanisms of proliferation of cancer signals, it is essential to change the mentality of certain farmers in the region of Boumerdes (Algeria) with regard to compliance with good harvesting practices. Optimal for picking olives, which is between the end of September and the end of October for the Chemelal variety. And finaly, the work should be extended to cover the sensory aspects of olive oil.

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