European Food Research and Technology (2024) 250:1443–1456
https://doi.org/10.1007/s00217-024-04475-2

ORIGINAL PAPER

Leaf mineral profiling and its correlation with oil physicochemical traits from four olive (*Olea europaea* L.) cultivars grown in Morocco as affected by olive ripening stages

Jamila Gagour[1] · Mohamed Ibourki[1,2] · Abderraouf El Antari[3] · El Hassan Sakar[4] · Rabha Aissa[5] · Angelo Maria Giuffrè[6] · Abdellatif Laknifli[1] · Said Gharby[1]

Received: 9 November 2023 / Revised: 10 January 2024 / Accepted: 13 January 2024 / Published online: 28 February 2024

Abstract

We investigated genotypic effects on the olive leaves mineral profile and its correlation with soil minerals. Likewise, olive oil extracted from four Mediterranean cultivars ('Arbequina', 'Haouzia', 'Menara', and 'Picholine Languedoc') at early and full ripening stages, was studied in terms of basic quality indices, fatty acids, sterols, pigments, and polyphenols. Our outcomes reveal important variations among cultivars and between ripening stages in terms of olive leaf elemental profiling and oil physicochemical traits, while there were no significant ($p < 0.05$) differences in soil mineral profiling. However, mineral profiling of leaves, basic quality indices, pigments, and polyphenols content of oils showed important inter-cultivar variations. Regarding fatty acid composition, oleic acid (C18:0) was the most abundant. For phytosterols profile, β-sitosterol was found to be the major phytosterol followed by campesterol. Olive oil from fully ripe fruits was marked by reduced chlorophylls (up to − 67.1%), carotenoids (up to − 68.73%) in 'Menara' and polyphenols (up to − 45.95%) in 'Picholine Languedoc', but an increase of total sterols (up to + 23.5%, 'Haouzia'). Likewise, saturated (SFA) and monounsaturated fatty acids (MUFA) tended to decrease (up to − 13.5% and − 6.44%, respectively) found in 'Menara'. However, 'Arbequina' had an inceased SFA (+ 7.35%) and MUFA (+ 8.62%). Polyunsaturated fatty acids tended to increase (up to + 41.98%, 'Menara') except for 'Picholine Languedoc' (− 7.92%). These outcomes were confirmed by principal component analysis with important positive and negative correlations among minerals and oil physicochemical traits. These results showed that the analyzed components could be considered as specific markers to discriminate the studied cultivars.

✉ Angelo Maria Giuffrè
amgiuffre@unirc.it

✉ Said Gharby
s.gharby@uiz.ac.ma

Jamila Gagour
jamilagagour1998@gmail.com

Mohamed Ibourki
mohamed.ibourki@um6p.ma

Abderraouf El Antari
a_elantari@yahoo.fr

El Hassan Sakar
e.sakar@uae.ac.ma

Rabha Aissa
r.aissa@uiz.ac.ma

Abdellatif Laknifli
a.laknifli@uiz.ac.ma

[1] Biotechnology, Analytical Sciences and Quality Control Team, Laboratory of Analysis Modeling, Engineering, Natural Substances and Environment, Polydisciplinary Faculty of Taroudant, University Ibn Zohr, Agadir, Morocco

[2] African Sustainable Agriculture Research Institute (ASARI), Mohammed VI Polytechnic University (UM6P), Laayoune, Morocco

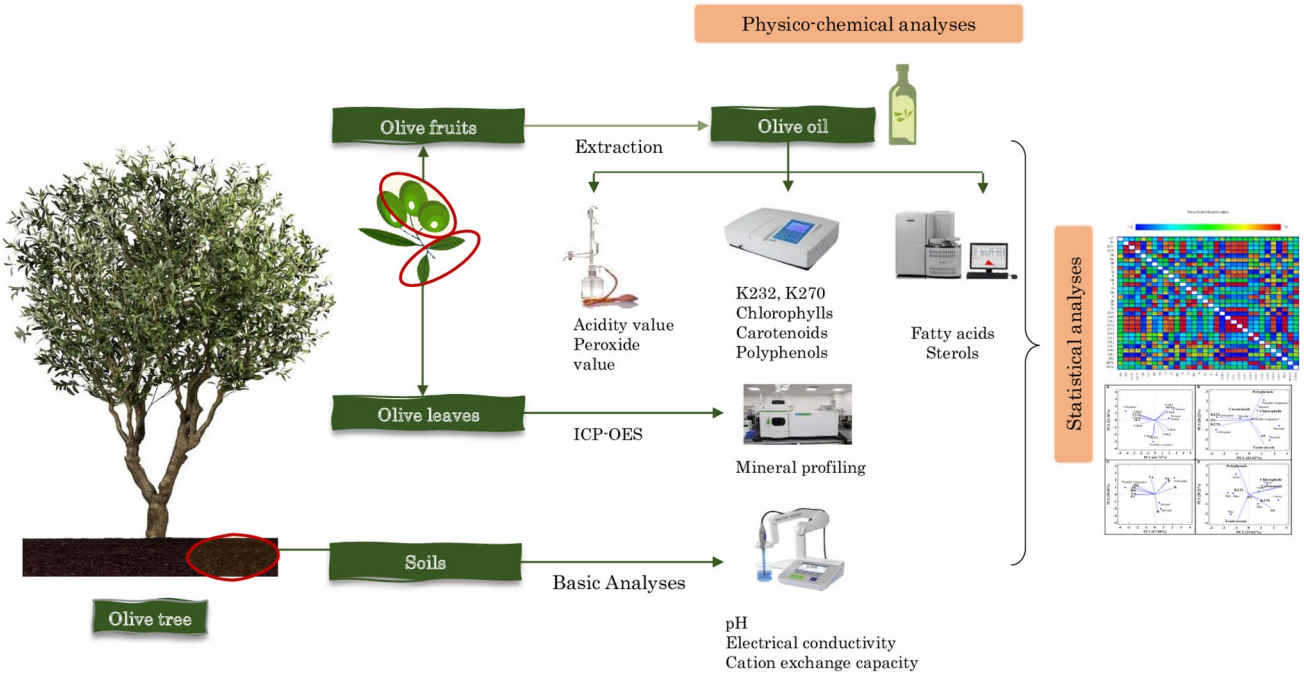
[3] Laboratory of Agro- Food Technology and Quality, Regional Center for Agronomic Research of Marrakech, National Institute of Agronomic Research (INRA), Marrakech, Morocco

[4] Laboratory of Biology, Ecology, and Health, FS, Abdelmalek Essaadi University, Tetouan, Morocco

[5] Bioprocesses and Environment Team, Superior School of Technology, Ibn-Zohr University, 80000 Agadir, Morocco

[6] Department AGRARIA, University of Studies "Mediterranea" of Reggio Calabria, 89124 Reggio Calabria, Italy

Graphical abstract



Keywords Olive cultivars · Oil quality · Leaf minerals · Ripening stage · Multivariate analysis

Introduction

Olive tree (*Olea europaea* L.) is an evergreen tree belonging to the Oleaceae family. About 11 million ha of olive groves exist in a total of 47 countries according to the International Olive Council (IOC) as discussed in Rallo et al. [1]. However, the Mediterranean basin is the main olive-growing area (over 97%) in the world [1]. Olives are used since about 6.000 years ago to produce table olives and olive oil, which is the main fat source in the Mediterranean diet [1, 2]. According to the IOC data, Spain is the first producer of olive oil worldwide (1,389,000 t) followed by Italy (273,500 t), and Greece (275,000 t). The global olive oil production witnessed a Moroccan contribution of 5% during the 2020/2021 crop campaign, ranking the country as the sixth-largest producer with an output of 160 000 tons. The 'Moroccan Picholine' cultivar stands as the predominant variety, constituting over 96% of the nation's genetic olive stock. Other cultivars produced in Morocco under Mediterranean breeding programs such as 'Picholine Languedoc,' 'Arbosana,' 'Haouzia,' 'Menara,' 'Meslala,' 'Arbequina,' and 'Picual' [3–5]. Like other Mediterranean countries, the Morocccan olive oil industry plays a pivotal socio-economic role and contributes to national development. Therefore, the Moroccan authorities have made extensive investments to promote this sector as a part of the implementation of the "Green Generation 2020–2030" strategy [6].

Olive oil fortifies the human diet owing to its interesting nutritional components namely polyphenolic compounds, phytosterols, tocopherols, and high monounsaturated fatty acids especially oleic acid [7]. These components are associated to numerous health benefits [2]. Olive oil is characterized by its unique organoleptic attributes and aroma compounds [8, 9]. Several clinical studies have demonstrated that olive oil has several beneficial effects on human health. Among them, antifungal, anti-inflammatory, antioxidant, antiulcerative, antibacterial, and antitumoral activities [8, 10, 11].There is a growing demand on olive oil and table olives as healthy foods leading to production increase of such products [1]. Olive leaves are also one of the most important valuable by-products of olive oil industry. Thanks to their great nutritional value, they serve for, a long time, as an animal feed [12]. Recently, they attract more attention owing to their composition rich in valuable bioactive compounds, particularly polyphenols and flavonoids [13–15]. This chemical composition richness makes olive oil distinguishable from other different vegetable oils. Olive oil bioactive compounds vary widely depending upon several factors such as cultivar, fruit ripening stage, growing region, extraction method, agronomic practices, environmental conditions (edaphic characteristics and climatic conditions), storage

time prior to oil extraction, among others [1, 2, 8, 16–20]. Several studies have been done to highlight effects of these factors on the chemical composition and quality of olive oil in different countries [2, 16, 18, 21–23]. To our knowledge, there is no detailed information on correlations among leaf mineral profiling and oil physicochemical traits in the main commercial olive cultivars grown in Morocco. Hence the novelty of this paper, which had as goals, (i) to assess of leaf mineral profiling and oil physicochemical traits in four olive cultivars grown in Morocco, (ii) to determine effects of ripening stage on some physicochemical traits of olive oil from these cultivars, and (iii) to evaluate correlations of leaf minerals and oil physicochemical traits.

Materials and methods

Plant material and sampling sites

The plant material used in this study consisted in four Mediterranean olive commercial cultivars widely grown in Morocco. The studied olive cultivars are 'Arbequina' (Spain), 'Menara' and 'Haouzia' (Morocco), and 'Picholine Languedoc' (France). Olives samples were hand picked around the canopy of healthy trees from the above mentioned cultivars at two ripening stages namely BBCH 80 (according to BBCH phenological scale) as the first ripening stage (fruit becoming light green or yellowish) and at the full ripening stage known as BBCH 89. Leaves and olives of the investigated cultivars were sampled in triplicate in the Saada experimental field, which belongs to the National Institute for Agronomic Research (INRA) located in the Marrakech-Safi region. Sampling was performed in the 2020/2021 crop season. Soil samples were also obtained from the same location.

Soil analysis

Soil samples were taken in 60 cm depth then dried at 40 °C and grounded into a powder to pass to a 2 mm sieve. Samples prepared were investigated for pH, electrical conductivity (EC), cation exchange capacity (CEC), organic matter (OM), minerals (K, Mg, Zn, Cu, Mn, Fe, and Na), nitrates, total nitrogen, and ammonium. Both pH and EC were determined using a laboratory meters for pH and conductivity (Mettler Toledo) according to [NF ISO 10390, 2005] [24], and [NF ISO 11265, 1994] [25], respectively. Organic carbon (OC) was determined following NF X 31-109 then (OM) was calculated by multiplying the total carbon by 1.724. Total nitrogen was determined using an elemental analyzer LECO FP 628. Minerals were determined according to NF X31-121 and NF X31-108 [26], using an inductively coupled plasma optical emission spectrometer (ICP-OES).

Leaf mineral analysis

Mineral's determination has been performed using an inductively coupled plasma optical emission spectrometer. A furnace muffle (Nabertherm) was used for calcination and then the obtained ash was digested using a concentrated nitric acid and oxygenated water. Total nitrogen determination was performed using an elemental analyzer LECO FP 628.

Oil extraction

After fruit harvest, for each studied ripening stage, the olive samples were immediately transported to the laboratory and oils were extracted according to the Abencor system.

Oil physicochemical traits

To characterize the extracted oils. A set of physicochemical traits were measured. These include basic quality indices, fatty acids and sterol profiling, pigments as well as polyphenols. These are very important since they define the classification of olive oil, its quality, and stability [4]. Acid value (AV), peroxide value (PV), and UV extinction coefficients (K232 and K270) were determined according to the European Commission Regulation EEC/2568/91 (2003) using a spectrophotometer double beam UV-6300PC VWR. AV was expressed as g/100 g of oleic acid. PV was expressed as milliequivalent of active oxygen per kilogram of oil (mEq O_2/kg oil) and UV extinction coefficients as the specific extinction of a 1% (w/v) solution of oil in cyclohexane. Fatty acid composition was determined following the ISO standard method [ISO 12966-2.2017] [27]. Briefly, fatty acids were converted into their corresponding fatty acid methyl esters (FAMEs) and then analyzed using a gas chromatography (Agilent-6890) coupled with a flame ionization detector (GC-FID). The capillary column CP-Wax 52CB (30 m × 250 µm i.d. 0.25 µm film thickness) was used. Helium was used as a carrier gas and the gas flow rate was set at 1 mL/min. The initial and final oven temperatures were 170 and 230 °C, respectively. The temperature gradient was 4 °C/min. Injector and detector temperatures were both set at 220 °C. The injection volume of the samples was 2 µL in a split mode (split ratio 1: 50). The results were expressed as the relative percentage of the area of each fatty acid peak. The sterol composition was determined according to the ISO standard [EN ISO 12228,2014] [28], using gas chromatography Varian 3800 instrument equipped with a VF-1 ms column (30 m & 0.25 mm id.) and using helium as a carrier gas (with a flow rate of 1.6 mL/min). Column temperature was isothermal at 270 °C, while the injector and detector temperatures were set at 300 °C. The jnjected volume was 1 mL. Data were processed using Varian Star Workstation v 6.30 (Varian Inc., Walnut Creek. CA., USA). Chlorophylls

and carotenoids were determined using a spectrophotometer according to the following equations: [Chlorophylls, mg kg^{-1}] = A670 × 106/613 × 100 × d and [Carotenoids, mg kg^{-1}] = A470 × 106/2000 × 100 × d. Where A is the absorbance, and d is the spectrophotometer cell thickness (1 cm). Chlorophylls and carotenoids levels were expressed as mg of pheophytin "a" and lutein per 100 g of oil, respectively [29]. Polyphenols were determined using a spectrophotometer at a wavelength of 725 nm. The isolation was performed by triple extraction of a solution of oil in hexane with a water/methanol mixture (60:40, v/v), then the Folin-Ciocalteu reagent was added [30].

Statistical analysis

The reported values represent mean values with standard deviations (SD, n = 3). Mean values separation was done using the Least Significant Difference (LSD) test at a 0.05 probability level. Data normality was checked using Shapiro–Wilk test. Principal component analysis (PCA) and Pearson correlations matrix were conducted based on the data mean values. All computations and analyses were carried out using Statgraphics XVII (Statpoint Technologies, Inc., Virginia, USA).

Results and discussion

Soil physicochemical properties

In this study, soil physicochemical parameters were determined for all sampling sites. Such paramters include pH, electrical conductivity (EC), organic matter (OM), total limestone (HCO_3), cation exchange capacity (CEC), ammonium nitrogen (N–NH_4), nitrate nitrogen (N–NO_3), and minerals. The obtained data are presented in Tables 1 and 2.

pH parameter serves as a critical indicator of nutrient bioavailability, encompassing both cations and anions in soil. It also influences microbial activity within the soil environment [31]. The obtained values of pH in this study were similar for all analyzed samples ($p < 0.05$), and show that all soils were alkaline (above 8). Our findings are Similar to those provesely reported (8.7) [32], but lower than that reported by [33]. Application of compost of two-phase olive mill waste on olive grove: effects on soil, olive fruit and olive oil quality. Electrical conductivity (EC) of the soil refers to the concentration of mineral salts dissolved in the soil. Higher EC means higher concentration of soluble salts in the soil. This will affect the amount of water and nutrients to be absorbed by the plant leading to its deterioration [34]. Our EC values varied between 0.21 and 0.51 and 51 revealing a low EC for all soils. Organic matter (OM) plays a major role in the global soil properties, in particular by influencing soil fertility. It contributes significantly to the buffering capacity of soil pH, and exerts an influence on various other soil characteristics such as soil structure, cation exchange capacity, nutrient turnover, nutrient retention capacity and soil stability [31]. The obtained orgnic matter (OM) values show little variation and ranged between 18.29 and 23.7%. CEC is a measure of soil ability to retain and blurt positively charged ions (Na, K, Ca, and Mg). This property has a key role since it influence the bioavailability of nutrients and pH [34]. CEC values obtained in this study ranged from 14.75 to 16.45 mol/kg. Ammonium nitrogen (N–NH_4) and nitrate

Table 1 Mean values of soil physicochemical parameters of the sampling sites

Site	pH	EC (ms/cm)	OM (%)	N (%)	TL (%)	CEC (mol/kg)	N–NO_3 (mg/kg)	N–NH_4 (mg/kg)
1	8.3 ± 0.61^a	0.21 ± 0.01^b	22.25 ± 1.65^a	1.68 ± 0.12^a	27.73 ± 2.04^b	15.00 ± 1.11^a	1.77 ± 0.13^a	0.38 ± 0.02^a
2	8.3 ± 0.61^a	0.23 ± 0.01^b	21.82 ± 1.62^a	1.67 ± 0.12^a	34.42 ± 2.54^a	14.75 ± 1.09^a	1.11 ± 0.08^b	0.48 ± 0.03^a
3	8.1 ± 0.58^a	0.51 ± 0.03^a	18.29 ± 1.35^a	1.59 ± 0.11^a	23.2 ± 1.71^b	14.80 ± 1.03^a	0.97 ± 0.07^b	0.40 ± 0.02^a
4	8.1 ± 0.56^a	0.40 ± 0.02^a	23.70 ± 1.75^a	1.71 ± 0.12^a	29.19 ± 2.16^b	16.45 ± 1.21^a	0.85 ± 0.06^b	0.41 ± 0.03^a

These parameters consist of pH, EC (electrical conductivity), OM (organic matter), TL (total limestone), CEC (Cation exchange capacity), N–NO_3 (nitrate), N–NH4 (ammonium). Within each column, values followed by the same letter are not significant different at $p < 0.05$

Table 2 Mineral composition of soils of the studied sites (sampling points)

Site	K (g/kg)	Mg (g/kg)	Cu (mg/kg)	Zn (mg/kg)	Mn (mg/kg)	Fe (mg/kg)	Na (g/kg)
1	0.36 ± 0.05^a	0.92 ± 0.17^a	9.33 ± 1.88^a	3.81 ± 0.11^a	24.5 ± 0.07^a	3.95 ± 0.07^b	0.09 ± 0.01^b
2	0.30 ± 0.07^a	0.79 ± 0.03^a	4.97 ± 0.04^b	2.65 ± 0.21^a	20.06 ± 0.05^b	4.04 ± 0.06^b	0.10 ± 0.04^b
3	0.34 ± 0.06^a	0.78 ± 0.03^a	5.52 ± 0.74^b	2.49 ± 0.16^a	24.22 ± 0.33^a	4.73 ± 0.35^a	0.15 ± 0.08^a
4	0.35 ± 0.06^a	0.9 ± 0.06^a	6.64 ± 0.19^b	3.51 ± 0.12^a	26.66 ± 0.27^a	5.04 ± 0.22^a	0.16 ± 0.08^a

The results are mean of three values followed by standard deviation (SD, n = 3). Within each column, values followed by the same letter are not significant different at $p < 0.05$

nitrogen (N–NO_3) show very similar content in soils and ranged from 0.85 to 1.77 mg/kg and from 0.38 to 0.48 mg/kg, respectively. Based on analysis of the parameters studied, the soils analyzed had almost similar chemical compositions.

Seven minerals were determined in the soils brought back from sampling areas. Soil element content of the four studied sites showed only moderate variations. The macro-elements namely K (0.30 ± 0.07–0.36 ± 0.05), Mg (0.78 ± 0.03–0.92 ± 0.17), and Na (0.09 ± 0.01–0.16 ± 0.08 g/kg) were found to be similar among samples with no statistical difference ($p < 0.05$) detected.. Similar trends were observed in the case of micro-elements namely Cu (4.97 ± 0.04–9.33 ± 1.88), Zn (2.65 ± 0.21–3.81 ± 0.11), Mn (20.06 ± 0.05–26.66 ± 0.27), and Fe (3.95 ± 0.07 to 5.04 ± 0.22 mg/kg). These results indicate no significant differences between the soils at the sampling sites, and their mineral content is therefore considered homogeneous [33].

Leaves mineral profiling

Mineral profiling of fully exptended and healthy leaves was performed for the four studied olive caultivars. The results reveal the presence of 10 distinct elements as indicated in Table 3.

Analyzing leaf nutrient levels is regarded as the most effective approach for assessing the nutritional status of trees. This practice is widely employed as it aids in identifying the necessary fertilization requirements for future applications [35]. The studied elements could be classified into two groups. Major elements namely P, K, Ca, and Mg and micro-elements (Cu, Mn, B, Fe, Zn, and Na). These elements play crucial roles in various metabolic processes and must exist within specific concentrations in plants. Deficiencies in these elements may lead to growth and yield issues, while excessive concentrations can have detrimental effects. The leaves mineral composition exhibited significant variations ($p < 0.05$) among different cultivars. Calcium (Ca) emerged as the predominant element across all cultivars, aligning with findings in published literature [36]. As compared to the other cultivars, 'Arbequina' leaves demonstrated the highest concentrations of three major elements: Ca (29,255.20 ± 2369), K (9480.20 ± 767.89), and P (and 1959.40 ± 158.70 mg/kg). Whereas 'Picholine Languedoc' leaves showed the highest concentrations concerning Mg (3021.71 ± 244 mg/kg) as compared to the remaining cultivars. 'Haouzia' leaves had the lowest Ca concentration (18,916.41 ± 1532) and P (978.84 ± 79 mg/kg) in comparison with the other cultivars. 'Picholine Languedoc' and 'Menara' leaves showed the lowest concentration of K (7088.36 ± 574) ang Mg (1948.8 ± 157 mg/kg), respectively. For all studied cultivars, the following order P < Mg < K < Ca was found for all macro-elements. Regarding micro-elements, 'Picholine Languedoc' leaves were characterized by a higher concentration of Fe (203 ± 16.44), Mn (143.65 ± 11.63), Zn (60.87 ± 4.93), and Cu (34.87 ± 2.82 mg/kg) compared to the other cultivars. 'Arbequina' leaves contained the lowest concentrations of Fe (127.19 ± 10) and Cu (5.90 ± 0.47 mg/kg), while those of 'Menara' showed the lowest levels of Mn (56.73 ± 4.59) and Zn (18.30 ± 1.48 mg/kg). The level of B was found to be very similar in all cultivars. 'Arbequina' leaves were characterized by a higher concentration of Na (168.83 ± 13.67 mg/kg).

Olive oil physicochemical traits

In order to evaluate the effect of genotype (cultivar) and fruit ripening stage (green/yellowish or fully ripe fruits) on the extracted vegetable oils, a set of quality indices were determined. They consist of acid value (AV), peroxide value (PV), extinction indices (K232, K270). Some purity criteria (fatty acids and sterols composition) were investigated and the obtained results are shown in Tables 4, 5, and 6.

Table 3 Mineral composition of olive leaves (mg/kg) of the studied cultivars

Mineral/cultivar	Arbequina	Menara	Picholine Languedoc	Haouzia
Ca	29,255.20 ± 2369^{a}	20,137.50 ± 1631^{c}	27,793.59 ± 2251^{b}	18,916.41 ± 1532^{d}
K	9480.20 ± 767.89^{a}	8275.33 ± 670^{b}	7088.36 ± 574^{d}	7867.12 ± 637^{c}
Mg	2243 ± 181^{b}	1948.80 ± 157^{d}	3021.71 ± 244^{a}	2052.33 ± 166^{c}
P	1959.40 ± 158.70^{a}	1059 ± 85.78^{c}	1067 ± 86.4^{b}	978.84 ± 79.00^{d}
Fe	127.19 ± 10^{d}	163.5 ± 13.25^{b}	203.00 ± 16.44^{a}	146.25 ± 11.84^{c}
Mn	57.00 ± 4.61c	56.73 ± 40.59^{c}	143.65 ± 11.63^{a}	60.56 ± 4.90^{b}
B	22.53 ± 1.85^{b}	24.40 ± 1.97^{a}	23.00 ± 1.86^{b}	25.33 ± 2.05^{a}
Zn	23.39 ± 1.89^{b}	18.30 ± 1.48^{d}	60.87 ± 4.93^{a}	20.47 ± 1.86^{c}
Cu	5.90 ± 0.47^{d}	23.77 ± 1.92^{b}	34.87 ± 2.82^{a}	8.00 ± 0.64^{c}
Na	168.83 ± 13.67^{a}	115.60 ± 9.36^{b}	103.55 ± 8.38^{c}	90.72 ± 7.34^{d}

The results are mean values followed by standard deviation (SD, n = 3). Within each line, values followed by the same letter are not significantly different at $p < 0.05$

Table 4 Quality indices acidity value (AV), peroxide value (PV) and extinction indices K232, K270) of olive oil

Ripening/cultivar	AV (g/100 g oleic acid)	PV (mEq O_2/kg oil)	K232	K270
Early ripe (BBCH 80)				
Picholine Languedoc	0.25 ± 0.15^{de}	1.10 ± 0.50^{ab}	1.3 ± 0.01^{d}	0.13 ± 0.02^{b}
Haouzia	1.08 ± 0.25^{a}	0.70 ± 0.20^{b}	1.01 ± 0.02^{g}	0.15 ± 0.01^{b}
Menara	0.78 ± 0.15^{c}	1.50 ± 0.10^{ab}	1.22 ± 0.02^{e}	0.16 ± 0.01^{b}
Arbequina	0.16 ± 0.12^{e}	2.10 ± 0.50^{ab}	1.73 ± 0.02^{a}	0.21 ± 0.02^{a}
Fully ripe (BBCH 89)				
Picholine Languedoc	0.80 ± 0.2^{bc}	1.50 ± 0.50^{ab}	1.4 ± 0.01^{c}	0.14 ± 0.02^{b}
Haouzia	0.67 ± 0.18^{cd}	1.00 ± 0.30^{b}	1.10 ± 0.01^{f}	0.14 ± 0.01^{b}
Menara	0.13 ± 0.10^{e}	1.70 ± 0.70^{ab}	1.51 ± 0.03^{b}	0.17 ± 0.02^{ab}
Arbequina	1.29 ± 0.20^{b}	2.50 ± 0.80^{a}	1.75 ± 0.01^{a}	0.21 ± 0.01^{a}
Standard (IOC)	< 0.8	< 20	< 2.5	< 0.02

The results are mean values of three replicates ± standard deviation (SD, n = 3). Within the same column, for each ripening stage, values followed by the same letter are not significantly different at 5% as a probability level

Table 5 Mean values of fatty acids of olive oils of different cultivars (g/100 g) according to fruit ripening stages (early ripe and fully ripe fruits)

	Picholine Languedoc		Haouzia		Menara		Arbequina		IOC, 2021
	Early ripe	Fully ripe	Early ripe	Fully ripe	Early ripe	Fully ripe	Early ripe	Fully ripe	
Myristic acid (C14:0)	0.01 ± 0.00	0.02 ± 0.00^{a}	0.01 ± 0.00	0.01 ± 0.00	0.01 ± 0.00	0.01 ± 0.00	0.01 ± 0.00	0.01 ± 0.00	< 0.03
Palmitic acid (C16:0)	12.8 ± 0.1^{c}	12.7 ± 0.1^{b}	11 ± 0.1^{e}	11 ± 0.1^{d}	11.7 ± 0.1^{d}	10.3 ± 0.1^{f}	18.0 ± 0.1^{b}	21.0 ± 0.1^{a}	7.5–20.0
Palmitoleic acid (C16:1)	0.7 ± 0.1^{c}	0.7 ± 0.0^{b}	0.7 ± 0.1^{c}	0.7 ± 0.1^{c}	0.8 ± 0.1^{c}	0.6 ± 0.1^{c}	1.7 ± 0.1^{b}	2.3 ± 0.1^{a}	0.3–3.5
Heptadecanoic acid (C17:0)	0.1 ± 0.1^{a}	0.04 ± 0.00^{b}	0.0 ± 0.1^{a}	0.0 ± 0.1^{a}	0.0 ± 0.1^{a}	0.3 ± 0.1^{a}	0.1 ± 0.1^{a}	0.1 ± 0.1^{a}	< 0.40
Heptadecenoic acid (C17:1)	0.1 ± 0.1^{b}	0.07 ± 0.00^{b}	0.1 ± 0.1^{b}	0.1 ± 0.1^{b}	0.1 ± 0.1^{b}	0.6 ± 0.1^{b}	0.2 ± 0.1^{b}	1.8 ± 0.1^{a}	< 0.60
Stearic acid (C18:0)	1.8 ± 0.1^{a}	1.69 ± 0.09^{b}	1.19 ± 0.1^{cd}	1 ± 0.1^{d}	2.8 ± 0.1^{a}	2.0 ± 0.1^{b}	1.7 ± 0.1^{bc}	0.15 ± 0.1^{e}	0.5- 5.0
Oleic acid (C18:1)	59.8 ± 0.1^{d}	59.5 ± 0.4^{b}	71.9 ± 0.1^{a}	67.3 ± 0.1^{b}	72.0 ± 0.1^{a}	71.7 ± 0.1^{a}	61.7 ± 0.1^{c}	54.0 ± 0.1^{e}	55.0–83.0
Linoleic acid (C18:2)	22.9 ± 0.1^{a}	23.5 ± 0.8^{a}	12.9 ± 0.1^{d}	17.7 ± 0.1^{g}	10.2 ± 0.1^{h}	13.9 ± 0.1^{d}	15.4 ± 0.1^{e}	18.5 ± 0.1^{c}	2.5–21.0
Linolenic acid (C18:3)	1.1 ± 0.1^{a}	1.04 ± 0.04^{a}	1 ± 0.1^{a}	0.9 ± 0.1^{a}	0.9 ± 0.1^{a}	0.8 ± 0.1^{a}	0.9 ± 0.1^{a}	0.8 ± 0.1^{a}	< 1.00
Arachidic acid (C20:0)	0.3 ± 0.1^{a}	0.2 ± 0.0^{ab}	0.3 ± 0.1^{a}	0.2 ± 0.1^{a}	0.3 ± 0.1^{a}	0.2 ± 0.1^{a}	0.6 ± 0.1^{a}	0.6 ± 0.1^{a}	< 0.60
Gadoleic acid (C20:1)	0.4 ± 0.1^{a}	0.34 ± 0.05^{a}	0.4 ± 0.1^{a}	0.30 ± 0.1^{a}	0.3 ± 0.1^{a}	0.3 ± 0.1^{a}	0.2 ± 0.1^{a}	0.2 ± 0.1^{a}	< 0.50
SFA*	15.0 ± 0.1^{c}	14.6 ± 0.1^{c}	13.1 ± 0.1^{d}	12.1 ± 0.1^{e}	14.8 ± 0.1^{c}	12.8 ± 0.1^{d}	20.4 ± 0.1^{b}	21.9 ± 0.1^{a}	
MUFA**	61.0 ± 0.1^{d}	60.3 ± 0.1^{d}	73.0 ± 0.1^{a}	68.3 ± 0.1^{b}	73.2 ± 0.1^{a}	73.2 ± 0.1^{a}	63.8 ± 0.1^{a}	58.3 ± 0.1^{e}	
PUFA***	24.0 ± 0.1^{b}	22.1 ± 0.1^{b}	13.1 ± 0.1^{g}	18.6 ± 0.1^{d}	11.1 ± 0.1^{h}	14.7 ± 0.1^{f}	16.1 ± 0.1^{e}	19.3 ± 0.1^{c}	

The results are presented as mean values followed by standard deviation (SD, n = 3). Within the same line, values followed by the same letter are not significantly different at 5% as a probability level

**SFA* Saturated fatty acids

***MUFA* Monounsaturated fatty acids

****PUFA* Polyunsaturated fatty acids

Basic quality indices

Acid value (AV) serves as a crucial quality index utilized for the classification of olive oil, as per the International Olive Council (IOC) guidelines [7]. In the case of extra virgin olive oil (EVOO), the upper limit for AV is established at 0.8 g/100 g oleic acid [7]. Wide variations ($p < 0.05$) were found among the investigated cultivars, the highest AV (1.08 ± 0.25 g/100 g) was found in 'Haouzia' oil at BBCH 80 (beginning of ripening), followed by oil (1.29 ± 0.20 g/100 g) from fully ripe fruits (BBCH89) of 'Arbequina'. These values exceed the limit for EVOO, the remaining values were much lower than IOC limit (0.8 g/100 g) [7]. The obtained results for 'Picholine Languedoc', 'Arbequina' and 'Menara' were close to those found by Gharby et al. in olive oil and Alwana oil [37].

Table 6 Mean values of phytosterols in olive oil (g/100 g) obtained from the studied cultivars according to ripening stage

	Picholine Languedoc		Haouzia		Menara		Arbequina	
	Early ripe	Fully ripe	Early ripe	Fully ripe	Early ripe	Fully ripe	Early ripe	Fully ripe
Cholesterol	0.2 ± 0.1^{a}	0.2 ± 0.1^{a}	0.1 ± 0.1^{a}	0.2 ± 0.1^{a}	ND*	0.2 ± 0.1^{a}	0.2 ± 0.1^{a}	0.1 ± 0.1^{a}
Campesterol	4.10 ± 0.1^{a}	3.18 ± 0.1^{c}	3.2 ± 0.1^{c}	2.8 ± 0.1^{d}	4.0 ± 0.1^{a}	2.4 ± 0.1^{e}	3.6 ± 0.1^{b}	3.1 ± 0.1^{c}
Stigmasterol	0.7 ± 0.1^{bcd}	0.5 ± 0.1^{de}	0.5 ± 0.1^{de}	0.6 ± 0.1^{cde}	0.9 ± 0.1^{b}	0.4 ± 0.1^{e}	0.8 ± 0.1^{cb}	1.3 ± 0.1^{a}
β-sitosterol	94.1 ± 0.5^{a}	94.9 ± 1.1^{a}	94.5 ± 0.7^{a}	94.7 ± 1.5^{a}	93.9 ± 0.1^{a}	94.3 ± 0.9^{a}	94.4 ± 0.5^{a}	94.2 ± 0.5^{a}
Delta-7-Stigmasterol	0.5 ± 0.1^{ab}	0.4 ± 0.1^{ab}	0.6 ± 0.1^{a}	0.1 ± 0.1^{c}	0.1 ± 0.0^{c}	0.3 ± 0.1^{cb}	$0,1 \pm 0.0^{c}$	0.6 ± 0.1^{a}
Delta-7-Avenasterol	0.3 ± 0.1^{a}	0.5 ± 0.1^{a}	0.4 ± 0.1^{a}	0.4 ± 0.1^{a}	0.4 ± 0.1^{a}	0.3 ± 0.1^{a}	0.3 ± 0.1^{a}	0.3 ± 0.1^{a}
Others	0.2 ± 0.1^{d}	0.3 ± 0.1^{d}	0.7 ± 0.1^{bc}	1.2 ± 0.1^{a}	0.9 ± 0.1^{ab}	1.1 ± 0.1^{a}	$0.6 \pm 0.1b^{c}$	$0.4 \pm 0.1c$
Total sterols (mg/100 g)	183.4 ± 10.5^{c}	196.4 ± 7.5^{b}	205.5 ± 8.5^{a}	253.8 ± 5.5^{d}	195.7 ± 10.5^{b}	206.7 ± 10.0^{a}	182.5 ± 7.5^{c}	195.5 ± 4.5^{b}

The results are presented as mean values followed by standard deviation (SD, n = 3). Within the same line, values followed by the same letter are not significantly different at 5% as a probability level

**ND* Not detected

Peroxide value (PV) corresponds to the hydroperoxide level and provides a measure of the early stages of lipids oxidation [38]. The obtained PV values for all cultivars at both ripening stages (BBCH80 and BBCH89) were found to vary slightly from 0.70 ± 0.20 to 2.50 ± 0.80 mEq O_2/kg oil which is very low compared to the IOC limit. The specific extinction coefficients at 232 nm (K232) and 270 nm (K270) are considered as indicators of primary and secondary oxidation products, respectively [39], All obtained results for K232 were found to be lower than the IOC limit (2.5). However, K270 values were higher than IOC limit (> 0.22). According to these findings, 'Picholine Languedoc' (BBCH80 and BBCH89), 'Haouzia' (BBCH89), 'Menara' (BBCH80 and BBCH89) and 'Arbequina' (BBCH80) oils could be considered as EVOO, and the effect of cultivar was of a lesser magnitude.

Fatty acid composition

Fatty acids constitute vital components of the saponifiable fraction within vegetable oils, delineating both their nutritional significance and stability, particularly in monounsaturated fatty acids (MUFA). Oleic acid, in particular, stands out as the predominant monounsaturated fatty acid in olive oil, accounting for a concentration range of 55–83 g/100 g [1, 37, 40–44].

In this study, fatty acid profile of oils of different cultivars was investigated (Table 5). The obtained data show that among eleven fatty acids determined, oleic acid was the most abundant in oils of all cultivars and ranged from 54.0 ± 0.1 to 72.0 ± 0.1 g/100 g. Similar results were reported by other authors in previous studies [45, 46], similar results were reported by other authors in previous studies [43, 44], and also in other vegetable oils [47]. Linoleic acid was the second and ranged from 10.2 ± 0.01 to 23.5 ± 0.8 g/100 g, followed by palmitic acid C16:0 from 10.3 ± 0.1 to 21.0 ± 0.1 g/100 g the same order was obtained for all oils. Except linoleic and linolenic acids found in both early ripening stage (BBCH80) and full ripening stage (BBCH89) of 'Picholine Languedoc' exceeding slightly the limits established by the IOC. All other fatty acids were found within IOC range values. Similar content of myristic, palmitic, palmitoleic, heptadecanoic, stearic, arachidic and gadoleic acids were found in 'Picholine Languedoc', 'Haouzia', and 'Menara' cultivars. The values found for 'Arbequina' for the same acids are relatively far. There were also important variations among the studied cultivars in terms of SFA, MUFA, and PUFA. Regardless of ripening stage, the Spanish cultivar 'Arbequina' was marked by higher level of SFA, while the greatest contents of MUFA and PUFA were found in Moroccan cultivars ('Houazia' and 'Menara') and French cultivar ('Picholine Languedoc'). Concerning the ripening stages, taking all cultivars together, early ripening stage (BBCH80) was marked by high SFA and MUFA for all cultivars except 'Arbequina'. In contrast, the best records of PUFA were reported in full ripening stage (BBCH89) for the studied cultivars except for 'Arbequina' where the picture was reflected.

Bioactive minor components

Phytosterols are considered as important molecules due to their biological properties and their determination is of major interest due to their antioxidant activities and health promoting properties. These compounds are also used to check authenticity of oils [42, 48]. Usually, tocopherols content found in olive oil varies from 10 to approximately 350 mg/kg [1]. Six phytosterols were investigated during this study and the obtained results are shown in Table 6.

Olive oil is known to be particularly rich in β-sitosterol [47]. Notably, β-sitosterol emerged as the predominant phytosterol in oils abtained from both green and ripe fruits, with

very similar contents were found in all samples (93.9 ± 0.1–94.7 ± 1.5 g/100 g). The high level of apparent β-sitosterol is particularly interesting in view of its role in the absorption of dietary cholesterol in the intestines. The results obtained for β-sitosterol app were in agreement with the results previously reported by other authors for olive oil [48]. Campesterol, the second most abundant sterol, exhibited variations among cultivars, ranging from 2.4 ± 0.1 to 4.1 ± 0.1 g/100 g. The other sterols were minor such as stigmasterol, cholesterol, and brassicasterol. Our results revealed an increase in total sterol content in ripe olive oil compared to green olive oil. Several studies have highlighted the inherent variations in sterol composition influenced by factors such as fruit ripening stages. For example, and in contrast to our conclusions [49], observed a decrease in the total sterol content of "Arbequina" VOO from 1775.9 to 1513.7 mg/kg during the ripening stages, with β-sitosterol following a similar decreasing trend, as [50], reported a decrease in total sterols from 1702 to 1682 mg/kg. Additionally, the cultivar and environmental conditions, as explored by [51], played a role, with a range spanning from 1490 to 1114 mg/kg. Thus, various factors contribute of the variation in these minor compounds. Consistent with these results, existing literature confirms the role of sterols as a discerning indicator of both the variety and degree of ripeness in virgin olive oils [52].

Olive oil natural antioxidants (chlorophylls, carotenoids and polyphenols) are important components of the unsaponifiable fraction in vegetable oils of compounds since they are involved in preventing lipid oxidation processes of oils and produce pleasant flavours [53]. Based on the data acquired (Table 7), notable variations were revealed among cultivars and also between ripening stages.. Irrespective of the specific cultivar, oils derived from green olives consistently demonstrated higher concentrations of pigments and polyphenols. Specifically, chlorophylls levels ranged from 10.1 ± 0.70 to 37.6 ± 1.97 mg/100 g, carotenoid content varied between 5.05 ± 0.25 and 16.15 ± 0.63 mg/100 g, and polyphenol content ranged from 60 ± 10 to 370 ± 10 µg/g. The wide variation in the polyphenol content of the cultivars studied agrees with the literature [54]. However, higher chlorophylls range is reported in our outcomes compared with those reported in [54].

Principal component analysis

PCA is widely performed as a multivariate statistical method to reduce data dimentionality in many fields such as pomological investigations, chemometrics, and food science, among others [55–60]. In this study. In our study, the first three principal components (PC) were retained since they explained over 92% of the total variance in our data. As can be seen in Fig. 1A, cultivars are separated according to PC1 (66.73%) and PC2 (31.50%). Indeed, 'Arbequina' interacted with higher records of SFA (mainly C16:0 and C17:0), 'Menara' was associated with great level of PUFA (C18:1), 'Haouzia' with C14:0, and finally 'Picholine Languedoc' was linked to high amounts of PUFA (mainly C18:2). As shown in Fig. 1B, 'Arbequina' was associated with high K232, K270, and PV on the negative side of PC1, while both 'Picholine Languedoc' and 'Menara' were marked by high levels of pigments (carotenoids and chlorophylls), polyphenols and finally 'Haouzia' interacted with AV and total sterols toward the PC1 positive side. Figure 1C shows distribution of cultivars according to leaf mineral profiling, 'Arbequina' was associated with high levels of Na, P, and K, while both 'Haouzia' and Menara' interacted with high level of B. In contrast, 'Picholine Languedoc' was associated with great amounts of Mg, Zn, Mn, Cu, and Fe. In Fig. 1D, both ripening stages BBCH 80 (early ripe) and BBCH 89 (fully ripe olives) were discriminated through the third principal component (PC3 = 15.11%). On its positive side, BBCH 80 (early ripe) was marked by higher levels of pigments (carotenoids and chlorophylls), polyphenols, SFA, and MUFA while BBCH 89 (fully ripe olives) interacted with high

Table 7 Mean values of pigments (chlorophylls and carotenoids) and polyphenols for the studied olive cultivars according to ripening stages

Ripening/cultivar	Chlorophylls (mg/100 g)	Carotenoids (mg/100 g)	Polyphenols (µg/g)
Early ripe (BBCH 80)			
Picholine Languedoc	20.3 ± 2.40^{c}	7.70 ± 0.70^{c}	370 ± 10.00^{a}
Haouzia	37.6 ± 1.97^{a}	13.15 ± 0.49^{b}	130 ± 30.00^{c}
Menara	30.7 ± 2.40^{b}	16.15 ± 0.63^{a}	240 ± 10.00^{b}
Arbequina	18.7 ± 1.69^{d}	14.00 ± 0.60^{b}	80 ± 10.00^{d}
Fully ripe (BBCH 89)			
Picholine Languedoc	10.7 ± 0.98^{b}	5.05 ± 0.25^{b}	200 ± 30.00^{b}
Haouzia	13.0 ± 0.70^{a}	5.25 ± 0.42^{b}	90 ± 00.00^{c}
Menara	10.1 ± 0.70^{b}	5.05 ± 0.28^{b}	220 ± 10.00^{a}
Arbequina	13.7 ± 1.71^{a}	10.50 ± 0.50^{a}	60 ± 10.00^{d}

The results are expressed as mean values followed by standard deviation SD (n = 3). Within each column, for each ripening stage, values followed by the same letter are not significantly different at $p < 0.05$

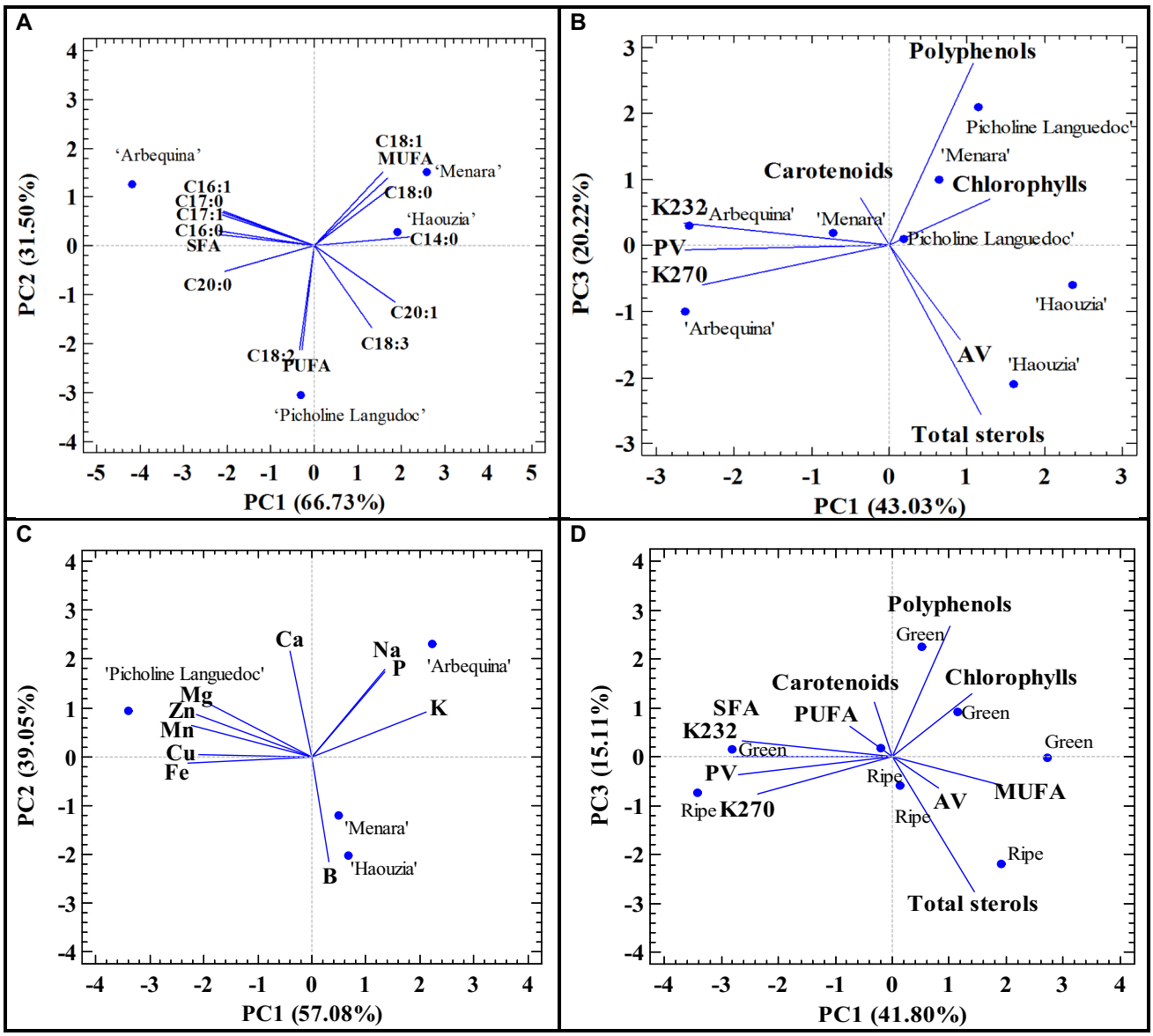


Fig. 1 Principal component (PC) projections for the first three PCs that most influenced leaf mineral content and olive oil physiochemical traits. Blue segments represent dependent variables, while points plotted are mean values. *AV* acid value, *PV* peroxide value, K232 and K270 are UV extinction coefficients, *SFA* saturated fatty acids, *MUFA* monounsaturated fatty acids, and *PUFA* polyunsaturated fatty acids. Green: early ripe olives and ripe: fully ripe olives

K270, AV, PV, PUFA, and total sterols on the PC3 negative side. The three components seem to be related mainly to genotypic variations among cultivars as well as olives ripening stage. These outcomes revealed by PCA confirmed those of mean values comparison reported in Tables 3, 4, 5, 6 and 7. Our results were consistent with published literature. In Fact, it was reported that with progress of ripening index, acid value (AV) increases but pigments and polyphenols tend to decrease. High AV values observed in advanced maturation stages could be attributed to an increase in enzymatic activity (lipolitic enzymes) as well to higher sensitivity to pathogenic infections [55, 57, 59–62]. Likewise, progressive decrease of pigments with olive maturation is ascribed mainly to reduction in the photosynthetic activity in agreement with previous findings from 'Moroccan Picholine' virgin olive oil [63].

Correlations analysis

Correlations matrix was carried on mean values to determine associations among the studied dependent variables and the results are summarized in Fig. 2. As can be seen in these outcomes, important positive and negative correlations were highlighted among different dependent variables. Among

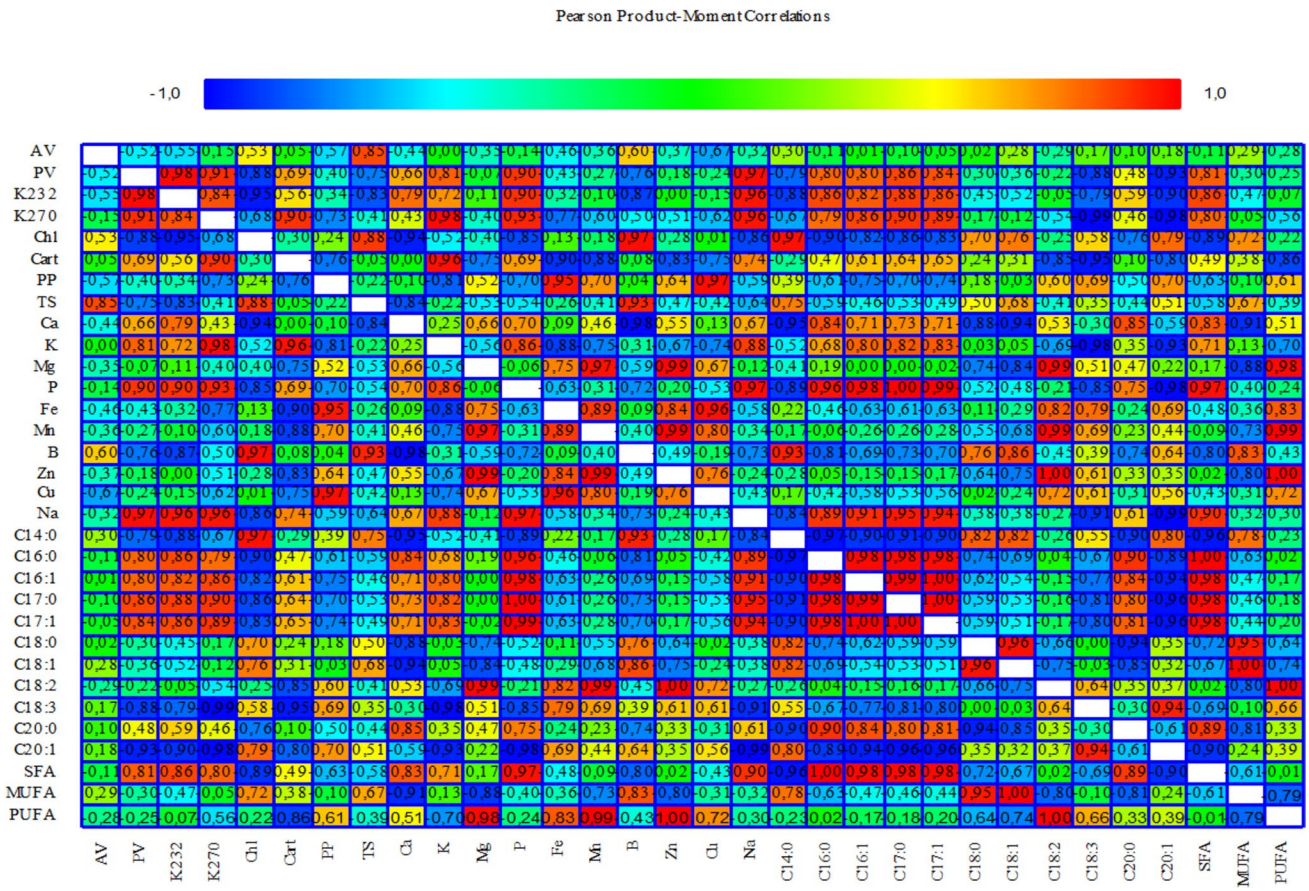


Fig. 2 Coefficients of correlations (Pearson correlation) among studied dependent variables. *AV* acid value, *PV* peroxide value, K232 and K270 are UV extinction coefficients, *Chl* chlorophylls, *Cart* carotenoids, *PP* polyphenols, *TS* total sterols, *SFA* saturated fatty acids, *MUFA* monounsaturated fatty acids, and *PUFA* polyunsaturated fatty acids

the most relevant positive correlations, there were K232 and K270, which were linked to each other on one hand and to PV on the other hand. K232 is a measure of primary oxidation products, which are instable and they are immediately converted into secondary oxidation products that absorb at 270 (K270), which explains the positive association between these two indices (K232 and K270). Likewise, several minerals were positively correlated to each other and also with pigments, polyphenols (PP), and fatty acids. Among them, K to carotenoids (cart) and P, Fe to PP, Mn to Fe and Mg, B to total sterols (TS) and chlorophylls (Chl), Zn to Mn, Fe, and Mg, Cu to Fe and PP, Na to P and K. Similar correlations were found in other works [64]. In a similar way, fatty acids correlated positively with some minerals. In fact, SFA correlated with Na, P, Ca, while MUFA were associated with B and Chl and finally PUFA were positively linked to Zn, Mn, Fe, and Mg. In contrast, important negative correlations were detected among several variables. In this regard, some minerals were negatively linked to each other such as Fe to K and B to Ca. Likewise, SFA were negatively correlated to B and Chl, MUFA were negatively associated with Zn, Mg, and Ca, while PUFA had negative correlations with K and Cart. As discussed in Ibourki et al. [56], positive correlations show a common source for the element pairs; however, negative correlations between element pairs confirm the independence of their sources. Strong positive and negative correlations could be used through simple/multiple regression models for prediction of an element from another for the element pairs. Gurel et al. studied correlations among fatty acids, total phenols, and minerals in soil, leaves, and fruit from 'Gemlik' cultivar [65]. These authors found similar results as reported in our work. As discussed in Ozdemir et al. [66], iron, copper and manganese are reported to be involved in lipid oxidation and linoleic biosynthesis. This maybe behind the negative associations among fatty acids and minerals.

Correlations among soil and leaf minerals

Correlations matrix among minerals in soil and leaves from the investigated cultivars are summarized in Fig. 3. Important negative and positive correlations were revealed among

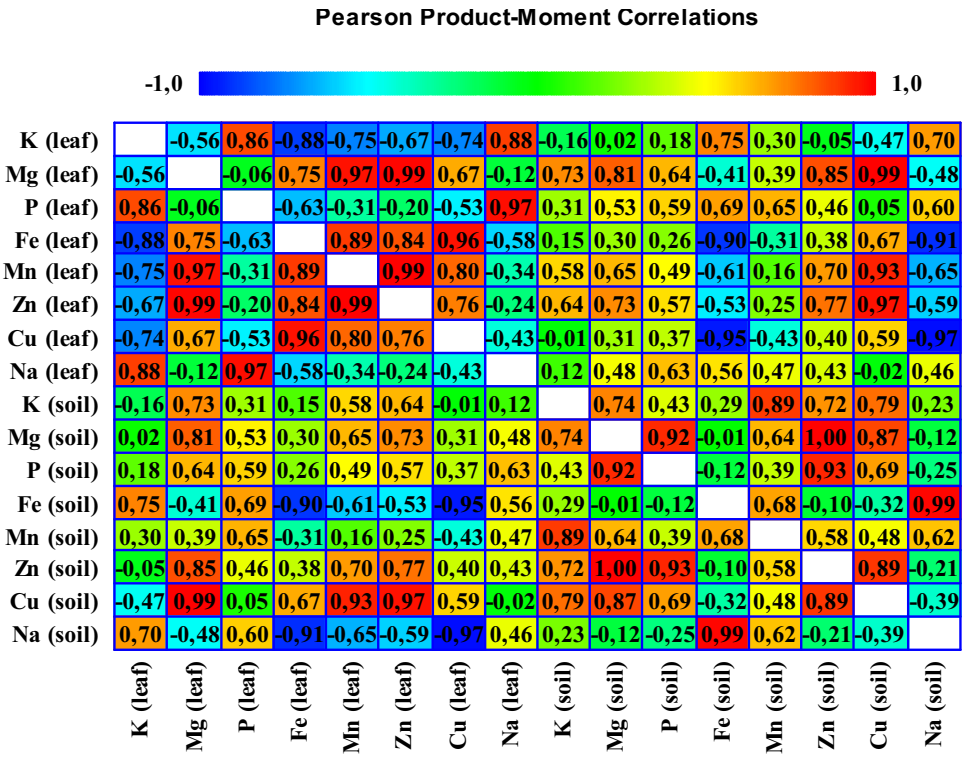


Fig. 3 Coefficients of correlations (Pearson correlation) among soil mineral and leaf mineral in all the studied cultivars

soil and leaf minerals. K (soil) was positively linked to Zn, Mg, Mn in leaves. Mg (soil) was positively associated with Zn, Mn, and Mg in leaves, while P (soil) was positively correlated to Na, Zn, P, and Mg in leaves. Fe (soil) was positively associated with Na, P, and K but negatively linked to Cu, Zn, Mn, and Mg in leaves. Mn (soil) was negatively linked to Cu but positively correlated with K and P in leaves. Zn (soil) was positively associated with Zn, Mn, and Mg in leaves. Cu (soil) was positively linked to Cu, Zn, Mn, Fe, and Mg in leaves but it was negatively associated with K (leaves). Na (soil) was positively correlated with Na, P, and K in leaves but it was negatively associated with Cu, Zn, Mn, Fe, and Mg in leaves. These outcomes were in agreement with published literature [64]. Increased contents of some minerals in soils results in high levels of such minerals in leaves owing to their absorption, explaining thus positives associations among some minerals. Negative correlations among some minerals in soil and leaves could be ascribed to their exchange during absorption or their low uptake by plant roots.

Conclusions

In conclusion, even the samples of the analyzed cultivars were from the same location and the obtained results for soils were similar. Several differences were detected in their chemical composition. Indeed, mineral composition of leaves and oil physiochemical traits showed important variations between cultivars. The same tendency was seen for chlorophylls, carotenoids and polyphenols. These differences could be explained by the effect of the cultivars and ripening stage. These results were better confirmed by principal compennt analysis. These findings show that the analyzed components seem to be an effective tool to discriminate between the cultivars, especially those related to oil purity. Ripening stage, at which olives harvested has to be considered as it determines overall oil quality parameters including basic indices, fatty acids, and minor bioactive components. However, a detailed volatile study seems to be required to complete the obtained results.

Acknowledgements We thank the Ibn Zohr University (UIZ), Agadir and National Institute of Agronomic Research, (INRA) Marrakech for their support and assistance in this work.

Author contributions Conceptualization, MI and SG; methodology, EHS and SG; software, JG, AEA, EHS and AMG; validation EHS and AMG; formal analysis, RA; investigation, RA; resources, AMG; data curation, RA and AL; writing—original draft preparation, SG and EHS; writing—review & editing, EHS, SG and AMG; visualization, EHS, SG and AMG; supervision, EHS, SG and AMG; project administration, AMG; funding acquisition, AMG. All authors have read and agreed to the published version of the manuscript.

Funding Open access funding provided by Università degli Studi Mediterranea di Reggio Calabria within the CRUI-CARE Agreement. This research was partially supported by: Distretto ad alta tecnologia agroindustriale della Calabria AGRIFOODTECH—PROGETTO PON03PE_00090_2. Sustainable models and new technologies for olives and olive oil.

Data availability Not applicable.

Declarations

Conflict of interest The authors declare no conflict of interest.

Compliance with ethics requirements This article does not present studies involving human or animal subjects.

References

1. Rallo L, Díez CM, Morales-Sillero A, Miho H, Priego-Capote F, Rallo P (2018) Quality of olives: a focus on agricultural preharvest factors. Sci Hortic 233:491–509. https://doi.org/10.1016/j.scienta.2017.12.034
2. Navajas-Porras B, Pérez-Burillo S, Morales-Pérez J, Rufián-Henares JA, Pastoriza S (2020) Relationship of quality parameters, antioxidant capacity and total phenolic content of EVOO with ripening state and olive variety. Food Chem 325:126926. https://doi.org/10.1016/j.foodchem.2020.126926
3. Sakar EH, Khtira A, Aalam Z, Zeroual A, Gagour J, Gharby S (2022) Variations in physicochemical characteristics of olive oil (Cv 'Moroccan Picholine') according to extraction technology as revealed by multivariate analysis. AgriEngineering 4:922–938. https://doi.org/10.3390/agriengineering4040059
4. Gagour J, Oubannin S, Bouzid HA, Bijla L, Moudden HE, Sakar EH, Koubachi J, Laknifli A, Gharby S (2022) Physicochemical characterization, kinetic parameters, shelf life and its prediction models of virgin olive oil from two cultivars ("Arbequina" and "Moroccan Picholine") grown in Morocco. OCL 29:39. https://doi.org/10.1051/ocl/2022033
5. IOC The World of Olive Oil Available online: https://www.internationaloliveoil.org/the-world-of-olive-oil/. Accessed on 18 Jan 2023
6. Azenzem R (2022) Oilseeds development in Morocco in the current international context. OCL 29:40. https://doi.org/10.1051/ocl/2022032
7. IOC Trade Standard Applying TO Olive Oils and And Olive Pomace Oils. COI/T.15/NC No 3/Rev. 16 June 2021
8. Bozdogan Konuskan D, Mungan B (2016) Effects of variety, maturation and growing region on chemical properties, fatty acid and sterol compositions of virgin olive oils. J Am Oil Chem Soc 93:1499–1508. https://doi.org/10.1007/s11746-016-2904-8
9. Ouni Y, Flamini G, Zarrouk M (2016) The chemical properties and volatile compounds of virgin olive oil from oueslati variety: influence of maturity stages in olives. J Am Oil Chem Soc 93:1265–1273. https://doi.org/10.1007/s11746-016-2863-0
10. Hatzakis E, Dagounakis G, Agiomyrgianaki A, Dais P (2010) A facile NMR method for the quantification of total, free and esterified sterols in virgin olive oil. Food Chem 122:346–352. https://doi.org/10.1016/j.foodchem.2010.02.043
11. Manai-Djebali H, Krichène D, Ouni Y, Gallardo L, Sánchez J, Osorio E, Daoud D, Guido F, Zarrouk M (2012) Chemical profiles of five minor olive oil varieties grown in central Tunisia. J Food Compos Anal 27:109–119. https://doi.org/10.1016/j.jfca.2012.04.010
12. Romero-García JM, Lama-Muñoz A, Rodríguez-Gutiérrez G, Moya M, Ruiz E, Fernández-Bolaños J, Castro E (2016) Obtaining sugars and natural antioxidants from olive leaves by steam-explosion. Food Chem 210:457–465. https://doi.org/10.1016/j.foodchem.2016.05.003
13. Guo Z, Jia X, Zheng Z, Lu X, Zheng Y, Zheng B, Xiao J (2018) Chemical composition and nutritional function of olive (*Olea europaea* L.): a review. Phytochem Rev 17:1091–1110. https://doi.org/10.1007/s11101-017-9526-0
14. Jilani H, Cilla A, Barberá R, Hamdi M (2016) Improved bioaccessibility and antioxidant capacity of olive leaf (*Olea europaea* L.) polyphenols through biosorption on saccharomyces cerevisiae. Ind Crops Prod 84:131–138. https://doi.org/10.1016/j.indcrop.2016.02.002
15. Özcan MM, Matthäus B (2017) A review: benefit and bioactive properties of olive (*Olea europaea* L.) leaves. Eur Food Res Technol 243:89–99. https://doi.org/10.1007/s00217-016-2726-9
16. Criado MN, Morelló JR, Motilva MJ, Romero MP (2004) Effect of growing area on pigment and phenolic fractions of virgin olive oils of the arbequina variety in Spain. J Amer Oil Chem Soc 81:633. https://doi.org/10.1007/s11746-004-954-z
17. Fernández-Cuesta A, León L, Velasco L, De la Rosa R (2013) Changes in squalene and sterols associated with olive maturation. Food Res Int 54:1885–1889. https://doi.org/10.1016/j.foodres.2013.07.049
18. de Mendoza MF, De Miguel Gordillo C, Marín Expóxito J, Sánchez Casas J, Martínez Cano M, Martín Vertedor D, Franco Baltasar MN (2013) Chemical composition of virgin olive oils according to the ripening in olives. Food Chem 141:2575–2581. https://doi.org/10.1016/j.foodchem.2013.05.074
19. Sicari V (2017) Antioxidant potential of extra virgin olive oils extracted from three different varieties cultivated in the Italian Province of Reggio Calabria. J Appl Bot Food Qual. https://doi.org/10.5073/JABFQ.2017.090.011
20. Yorulmaz A, Yavuz H, Tekin A (2014) Characterization of Turkish olive oils by triacylglycerol structures and sterol profiles. J Am Oil Chem Soc 91:2077–2090. https://doi.org/10.1007/s11746-014-2554-7
21. Cetinkaya H, Koc M, Kulak M (2016) Monitoring of mineral and polyphenol content in olive leaves under drought conditions: application chemometric techniques. Ind Crops Prod 88:78–84. https://doi.org/10.1016/j.indcrop.2016.01.005
22. Fernández-Poyatos MDP, Ruiz-Medina A, Llorent-Martínez EJ (2021) Phytochemical profile and mineral content of royal variety olive fruits influence of the ripening stage. J Food Compos Anal 95:103671. https://doi.org/10.1016/j.jfca.2020.103671
23. Martínez-Navarro ME, Cebrián-Tarancón C, Alonso GL, Salinas MR (2021) Determination of the variability of bioactive compounds and minerals in olive leaf along an agronomic cycle. Agronomy 11:2447. https://doi.org/10.3390/agronomy11122447
24. ISO 10390:2005 Soil quality—determination of pH Available online: https://www.iso.org/standard/40879.html. Accessed on 16 Jan 2023.
25. ISO 11265:1994 Soil quality—determination of the specific electrical conductivity Available online: https://www.iso.org/standard/19243.html. Accessed on 16 Jan 2023
26. NF X31–108 Soil quality, determination of ammonium acetate extractable Ca++, Mg++, K+ and Na+ cations - agitation

method. Available online: https://infostore.saiglobal.com/en-us/standards/iso-11263-1994-r2016--603116_saig_iso_iso_1381356/. Accessed on 2 Jan 2023
27. ISO 12966-2 Animal and vegetable fats and oils—Gas chromatography of fatty acid methyl esters—Part 2: preparation of methyl esters of fatty acids Available online: https://www.iso.org/cms/render/live/en/sites/isoorg/contents/data/standard/07/21/72142.html. Accessed on 7 Oct 2022
28. ISO 12228-1 Determination of individual and total sterols contents—gas chromatographic method — Part 1: animal and vegetable fats and oils available online: https://www.iso.org/standard/60248.html. Accessed on 30 Nov 2022
29. Gagour J, Ahmed MN, Bouzid HA, Oubannin S, Bijla L, Ibourki M, Hajib A, Koubachi J, Harhar H, Gharby S (2022) Proximate composition, physicochemical, and lipids profiling and elemental profiling of rapeseed (*Brassica napus* L.) and sunflower (*Helianthus annuus* L.) grown in Morocco. Evid-Based Complement Altern Med 2022:1–12. https://doi.org/10.1155/2022/3505943
30. Chatoui K, Harhar H, El Kamli T, Tabyaoui M (2020) Chemical composition and antioxidant capacity of *Lepidium sativum* seeds from four regions of Morocco. Evid-Based Complement Altern Med 2020:e7302727. https://doi.org/10.1155/2020/7302727
31. Ennaji W, Barakat A, El Baghdadi M, Oumenskou H, Aadraoui M, Karroum LA, Hilali A (2018) GIS-based multi-criteria land suitability analysis for sustainable agriculture in the Northeast Area of Tadla Plain (Morocco). J Earth Syst Sci 127:79. https://doi.org/10.1007/s12040-018-0980-x
32. Fernández-Hernández A, Roig A, Serramiá N, Civantos CG-O, Sánchez-Monedero MA (2014) Application of compost of two-phase olive mill waste on olive grove: effects on soil, olive fruit and olive oil quality. Waste Manage 34:1139–1147. https://doi.org/10.1016/j.wasman.2014.03.027
33. Damak F, Bougi MSM, Araoka D, Baba K, Furuya M, Ksibi M, Tamura K (2021) Soil geochemistry, edaphic and climatic characteristics as components of Tunisian Olive Terroirs: relationship with the multielemental composition of olive oils for their geographical traceability. Euro-Mediterr J Environ Integr 6:37. https://doi.org/10.1007/s41207-021-00241-y
34. Barakat A, Ennaji W, El Jazouli A, Amediaz R, Touhami F (2017) Multivariate analysis and GIS-based soil suitability diagnosis for sustainable intensive agriculture in Beni-Moussa irrigated subperimeter (Tadla Plain, Morocco). Model Earth Syst Environ 3:3. https://doi.org/10.1007/s40808-017-0272-5
35. Fernández-Escobar R, Moreno R, Garcıa-Creus M (1999) Seasonal changes of mineral nutrients in olive leaves during the alternate-bearing cycle. Sci Hortic 82:25–45. https://doi.org/10.1016/S0304-4238(99)00045-X
36. Bahloul N, Kechaou N, Mihoubi NB (2014) Comparative investigation of minerals, chlorophylls contents, fatty acid composition and thermal profiles of olive leaves (*Olea europeae* L.) as by-product. Grasas Aceites 65:e035. https://doi.org/10.3989/gya.0102141
37. Gharby S, Harhar H, Farssi M, Taleb AA, Guillaume D, Laknifli A (2018) Influence of roasting olive fruit on the chemical composition and polycyclic aromatic hydrocarbon content of olive oil. OCL 25:A303. https://doi.org/10.1051/ocl/2018013
38. Harhar H, Gharby S, El Idrissi Y, Pioch D, Matthäus B, Charrouf Z, Tabyaoui M (2019) Effect of maturity stage on the chemical composition of argan fruit pulp. OCL 26:15. https://doi.org/10.1051/ocl/2019012
39. Ghanbari Shendi E, Sivri Ozay D, Ozkaya MT (2020) Effects of filtration process on the minor constituents and oxidative stability of virgin olive oil during 24 months storage time. OCL 27:37. https://doi.org/10.1051/ocl/2020030
40. Al-Bachir M, Sahloul H (2017) Fatty acid profile of olive oil extracted from irradiated and non-irradiated olive fruits. Int J Food Prop 20:2550–2558. https://doi.org/10.1080/10942912.2016.1243557
41. Gharby S, Harhar H, Bouzoubaa Z, Asdadi A, El Yadini A, Charrouf Z (2017) Chemical characterization and oxidative stability of seeds and oil of sesame grown in Morocco. J Saudi Soc Agric Sci 16:105–111. https://doi.org/10.1016/j.jssas.2015.03.004
42. Gharby S, Hajib A, Ibourki M, Sakar EH, Nounah I, Moudden HE, Elibrahimi M, Harhar H (2021) Induced changes in olive oil subjected to various chemical refining steps: a comparative study of quality indices, fatty acids, bioactive minor components, and oxidation stability kinetic parameters. Chem Data Collect 33:100702. https://doi.org/10.1016/j.cdc.2021.100702
43. Ibourki M, Azouguigh F, Jadouali SM, Sakar EH, Bijla L, Majourhat K, Gharby S, Laknifli A (2021) Physical fruit traits, nutritional composition, and seed oil fatty acids profiling in the main date palm (*Phoenix dactylifera* L.) varieties grown in Morocco. J Food Qual 2021:1–12. https://doi.org/10.1155/2021/5138043
44. El Antari A, Sikaoui L (2022) Catalog of cultivated and indigenous olive varieties of Morocco. National Institute for Agronomic Research (INRA) Editions, Rabat, Morocco (INRA), p 76, ISBN: 978-9920-787-178. https://www.inra.org.ma/fr/content/catalogue-des-varietes-dolivier-cultivees-et-autochtones-dumaroc
45. Monfreda M, Gobbi L, Grippa A (2012) Blends of olive oil and sunflower oil: characterisation and olive oil quantification using fatty acid composition and chemometric tools. Food Chem 134:2283–2290. https://doi.org/10.1016/j.foodchem.2012.03.122
46. Rouina YB, Zouari M, Zouari N, Rouina BB, Bouaziz M (2020) Olive tree (*Olea europaea* L. Cv. Zelmati) grown in hot desert climate: physio-biochemical responses and olive oil quality. Sci Hortic 261:108915. https://doi.org/10.1016/j.scienta.2019.108915
47. Rękas A, Wroniak M, Krygier K (2015) Effects of different roasting conditions on the nutritional value and oxidative stability of high-oleic and yellow-seeded Brassica napus oils. Grasas Aceites 66:e092. https://doi.org/10.3989/gya.1299142
48. Taoufik F, Zine S, El Hadek M, Idrissi Hassani LM, Gharby S, Harhar H, Matthäus B (2015) Oil content and main constituents of cactus seed oils opuntia ficus indica of different origin in Morocco. MNM 8:85–92. https://doi.org/10.3233/MNM-150036
49. Ghreishi Rad SA, Jalili M, Ansari F, Rashidi Nodeh H, Rashidi L (2023) Maturity impact on physicochemical composition and polyphenol properties of extra virgin olive oils obtained from Manzanilla, Arbequina, and Koroneiki Varieties in Iran. Food Sci Nutr. https://doi.org/10.1002/fsn3.3497
50. Rey-Giménez R, Vázquez Ayala S, Laya Reig D, Sánchez-Gimeno AC (2023) Chemometric and physico-chemical characterization of fruit and olive oils from autochthonous cultivars grown in Aragon Spain. Foods 12:803. https://doi.org/10.3390/foods12040803
51. Rey-Giménez R, Sánchez-Gimeno AC (2023) Effect of cultivar and environment on chemical composition and geographical traceability of Spanish olive oils. JAOCS J Am Oil Chem Soc. https://doi.org/10.1002/aocs.12774
52. Lukić M, Lukić I, Krapac M, Sladonja B, Piližota V (2013) Sterols and triterpene diols in olive oil as indicators of variety and degree of ripening. Food Chem 136:251–258. https://doi.org/10.1016/j.foodchem.2012.08.005
53. Maqsood S, Benjakul S, Abushelaibi A, Alam A (2014) Phenolic compounds and plant phenolic extracts as natural antioxidants in prevention of lipid oxidation in seafood: a detailed review: polyphenols: antioxidants in seafood. Compr Rev Food Sci Food Saf 13:1125–1140. https://doi.org/10.1111/1541-4337.12106
54. Hannachi H, Nasri N, Elfalleh W, Tlili N, Ferchichi A, Msallem M (2013) Fatty acids, sterols, polyphenols, and chlorophylls of olive oils obtained from Tunisian wild olive trees (*Olea europaea* L.

Var. Sylvestris). Int J Food Properties 16:1271–1283. https://doi.org/10.1080/10942912.2011.584201
55. Bijla L, Ibourki M, Bouzid HA, Sakar EH, Aissa R, Laknifli A, Gharby S (2022) Proximate composition, antioxidant activity, mineral and lipid profiling of spent coffee grounds collected in Morocco reveal a great potential of valorization. Waste Biomass Valor. https://doi.org/10.1007/s12649-022-01808-8
56. Ibourki M, Ait Bouzid H, Bijla L, Sakar EH, Asdadi A, Laknifli A, El Hammadi A, Gharby S (2022) Mineral profiling of twenty wild and cultivated aromatic and medicinal plants growing in Morocco. Biol Trace Elem Res. https://doi.org/10.1007/s12011-021-03062-w
57. Khattabi D, Sakar EH, Louahlia S (2021) Flag leaf tolerance study in moroccan barley (*Hordeum vulgare* L.) varieties submitted to a severe salt stress. Biointerface Res Appl Chem 12:2787–2799. https://doi.org/10.33263/BRIAC123.27872799
58. Zeroual A, Sakar EH, Eloutassi N, Mahjoubi F, Chaouch M, Chaqroune A (2021) Phytochemical profiling of essential oils isolated using hydrodistillation and microwave methods and characterization of some nutrients in *Origanum compactum* benth from Central-Northern Morocco. Biointerface Res App Chem 11:9358–9371. https://doi.org/10.33263/BRIAC112.93589371
59. Zeroual A, Sakar EH, Ibourki M, Bijla L, Ainane A, Mahjoubi F, Chaouch M, Gharby S, Chaqroune A, Ainane T (2021) Phytochemical screening and mineral profiling of wild and cultivated rosemary (*Rosmarinus Officinalis* L.) from Taounate Region (Northern Morocco). Pharmacol Online 22:576–582
60. El Bakali I, Sakar EH, Boutahar A, Kadiri M, Merzouki A (2022) A comparative phytochemical profiling of essential oils isolated from three hemp (*Cannabis sativa* L.) cultivars grown in Central-Northern Morocco. Biocatal Agric Biotechnol 42:102327. https://doi.org/10.1016/j.bcab.2022.102327
61. Ibourki M, Ait Bouzid H, Bijla L, Aissa R, Sakar EH, Ainane T, Gharby S, El Hammadi A (2022) Physical fruit traits, proximate composition, fatty acid and elemental profiling of almond [*Prunus Dulcis* Mill .DA Webb] kernels from ten genotypes grown in Southern Morocco. OCL 29:9. https://doi.org/10.1051/ocl/2022002
62. Zeroual A, Sakar EH, Eloutassi N, Mahjoubi F, Chaouch M, Chaqroune A (2020) Wild Chamomile [*Cladanthus Mixtus* (L.) Chevall] collected from Central-Northern Morocco: phytochemical profiling, antioxidant, and antimicrobial activities. Biointerface Res Appl Chem 11:11440–11457. https://doi.org/10.33263/BRIAC114.1144011457
63. El Yamani M, Sakar EH, Boussakouran A, Rharrabti Y (2020) Influence of ripening index and water regime on the yield and quality of "Moroccan Picholine" virgin olive oil. OCL 27:19. https://doi.org/10.1051/ocl/2020015
64. Boukyoud Z, Ibourki M, Gharby S, Sakar EH, Bijla L, Atifi H, Matthäus B, Laknifli A, Charrouf Z (2021) Can the water quality influence the chemical composition, sensory properties, and oxidative stability of traditionally extracted argan oil? MNM 14:383–399. https://doi.org/10.3233/MNM-210005
65. Gurel S, Basar H, Sahan Y (2014) Antioxidative properties of olive fruits (*Olea europaea* L.) from 'Gemlik' variety and relationship with soil properties and mineral composition. Oxid Commun 4:985–1004
66. Bolek S, Ozdemir M (2017) Optimization of roasting conditions of pistacia terebinthus in a fluidized bed roaster. LWT 80:67–75. https://doi.org/10.1016/j.lwt.2017.02.007