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Physicochemical characterization, kinetic parameters, shelf life and its prediction models of virgin olive oil from two cultivars ("Arbequina" and "Moroccan Picholine") grown in Morocco

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Abstract – This works aimed at investigating shelf-life behavior of extra virgin olive oil (EVOO) extracted from two cultivars "Arbequina" and "Moroccan Picholine" as a function of storage time (8 weeks) at accelerated conditions (60 °C). Our outcomes revealed significant variations between EVOOs from both cultivars in terms of the investigated physicochemical characteristics. These were also affected by storage time and temperature except for fatty acids, for which storage time had no significant effects. While the changes in tocopherols showed a drastic reduction ranging from 48.18 ("Moroccan Picholine") to 62.62% ("Arbequina"). Indeed, the changes of quality indices showed a linear increase. Moreover, "Arbequina" oil was the first to exceed the established upper limits for EVOO label. An increase in oxidation rate was observed with increasing temperature when oils were oxidized at six elevated temperatures (373, 383, 393, 403, 413 and 423 °K) under Rancimat test conditions. The natural logarithms of the kinetic rate constant varied linearly with respect to temperature, with temperature coefficient (T_{Coeff}) ranging from 7.31×10^{-2} in "Arbequina" to $7.51 \times 10^{-2}\,K^{-1}$ found in "Moroccan Picholine". This had higher oxidative stability and shelf life as compared to "Arbequina". These outcomes were confirmed by kinetic parameters of oxidative stability including reaction rate constant as well as Arrhenius equation and thermodynamic parameters.

Keywords: accelerated storage / Arrhenius equation / "Moroccan Picholine" / oil stability index / shelf life

Résumé – Caractérisation physicochimique, paramètres cinétiques, durée de vie et modèles de prédiction de l'huile d'olive vierge provenant de deux cultivars (« Arbequina » et « Picholine marocaine ») cultivés au Maroc. L'objectif de ce travail est d'étudier le comportement de la durée de vie des huiles d'olive vierges extra (HOVE) extraites de deux cultivars (« Arbequina » et « Picholine marocaine »), en fonction du temps de stockage (8 semaines) dans des conditions accélérées (60 °C). Nos résultats ont révélé des variations significatives entre les HOVE des deux cultivars en termes de caractéristiques physicochimiques étudiées. Celles-ci ont également été affectées par le temps et la température de stockage, sauf pour les acides gras, pour lesquels le temps de stockage n'a eu aucun effet significatif. Alors que les changements des tocophérols ont montré une réduction drastique allant de 48,18 (« Picholine Marocaine ») à 62,62 % (« Arbequina »). En effet, les changements des indices de qualité ont montré une augmentation linéaire. En outre l'huile « Arbequina » a été la première huile qui a dépassé les limites supérieures établies pour le label HOVE. Une augmentation du taux d'oxydation a été observée avec l'augmentation de la température lorsque les huiles ont été soumises à six températures élevées (373, 383, 393, 403, 413 et 423 °K) sous Rancimat. Les logarithmes naturels de la constante de vitesse cinétique ont varié linéairement par rapport à la température, avec un coefficient de température (T_{Coeff}) allant de $7{,}31 \times 10^{-2}\,K^{-1}$ pour « Arbequina » à $7{,}51 \times 10^{-2}\,K^{-1}$ dans le cas de la « Picholine Marocaine »). Celle-ci a

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eu une stabilité oxydative et une durée de vie plus élevées par rapport à « Arbequina ». Ces résultats ont été confirmés par les paramètres cinétiques de la stabilité oxydative, y compris la constante de vitesse de réaction ainsi que les paramètres de l'équation d'Arrhenius et thermodynamiques.

Mots clés : stockage accéléré / équation d'Arrhenius / « picholine marocaine » / indice de stabilité de l'huile / durée de conservation

1 Introduction

Olive tree (*Olea europaea* L.) belongs to the oldest cultivated trees in the world. It stands as a symbol of friendship and peace between nations (Uylaser and Yildiz, 2014). It stands also as the main and most valuable fruit of the Mediterranean basin (Gharby *et al.*, 2016a). More than 11 million hectares of olive trees are cultivated worldwide, it spreads over 5 continents in almost 50 countries (Kiritsakis *et al.*, 2020). However, Mediterranean basin represents olive preferred area with over 90% of the world's olive groves. In this regard, in Morocco, olive tree occupies the largest arboricultural land area in the country, covering an expanse of about 1 Mha, with a production rate of about 1,500,000 tons of oil per year (Elgadi *et al.*, 2021; Hilali *et al.*, 2021). This makes Morocco in sixth rank in the world production of olive oil, behind the European Union, Tunisia, and Turkey (El Yamani *et al.*, 2022). Olive cultivation holds a crucial socio-economic role in the national and regional agricultural development. For these considerations, Moroccan government has deployed substantial efforts to strengthen Moroccan olive sector following the adoption of the "Green Morocco Plan" strategy, owing to its ecological, economic, and cultural importance (Gharby *et al.*, 2013).

There is an important diversity in terms of olive cultivars with an outstanding phenotypic diversity (Laaribi *et al.*, 2014). Morocco's climatic conditions have led to the spread of different cultivars across the whole national territory, due to its adaptive capacity to various bioclimatic stages, ranging from mountainous areas to arid and Saharan zones (El Qarnifa *et al.*, 2019; Zaroual *et al.*, 2021). The predominant cultivar in Morocco is the "Moroccan Picholine" (locally known as "Zeitoun Beldi") representing over 90% of Moroccan olive groves (Mansouri *et al.*, 2013; Harhar *et al.*, 2018). Different cultivars such as "Arbequina" and "Arbosana" have also been introduced into the central regions of the country (Zaroual *et al.*, 2021).

The importance of olive oil comes from its increasing consumption worldwide, thanks to its valuable nutritional properties compared to other vegetable oils (Dabbou *et al.*, 2011). Likewise, olive oil is resistant to oxidation owing to its high content of monounsaturated fatty acids (MUFA) and antioxidants (El Qarnifa *et al.*, 2019). In addition, it is appreciated by consumers thanks to its health benefits and pleasant, distinct flavor, resulting from a complex mixture of volatile compounds (Elgadi *et al.*, 2021). It is also a rich source of bioactive compounds including phenols, tocopherols, sterols, phospholipids, waxes, squalene (SQ), and other hydrocarbons constituting the unsaponifiable fraction (Kiritsakis *et al.*, 2020). Composition of this natural juice varies widely depending not only on environmental, agronomic, cultural, and technological factors, but also on genotype of the olives (Gharby *et al.*, 2013; Kiritsakis *et al.*, 2020).

Lipid oxidation is one of the most critical factors that can cause deterioration of olive oil quality (Farhoosh and Hoseini-Yazdi, 2014a). Despite olive oil is thought to be resistant to oxidation due to its low polyunsaturated fatty acid content and the presence of natural antioxidants. Nevertheless, like other vegetable oils, the effects of post-harvest and storage conditions promote the gradual oxidation of lipids, thus making olive oil susceptible to oxidation (Morales and Przybylski, 2000; El Yamani *et al.*, 2022). Oxidation leads directly to the formation of volatile products, which change not only the initial flavor, but also reduce the nutritional quality and may lead to the formation of toxic products, thus reducing the shelf life of the oil (Stefanoudaki *et al.*, 2010; Gharby, 2022).

To our knowledge, there are no detailed studies regarding the behavior of shelf life and its modelling under storage in olive oil from both cultivars "Moroccan Picholine" and "Arbequina" widely growing in Moroccan olive groves. Hence originality of this work. This aimed at (i) investigating and comparing olive oil physicochemical properties of these cultivars grown in Morocco, (ii) exploring storage effect on such physicochemical properties under accelerated conditions (60 °C), and (iii) addressing effect of temperature on kinetic parameters, rate constants, and shelf life.

2 Materiel and methods

2.1 Olive oil sampling

This study was conducted on olive oil sampled separately from "Arbequina" and "Moroccan Picholine" cultivars with three independent replicates for each cultivar. Sampling was carried out from a 3-phase extraction system (2020 crop season). At the moment of extraction, olives were at olives were at 5–6 ripening index, reaching the cultivar typical color, being turgid and suitable for oil extraction as described in Sakar *et al.* (2022). To avoid oxidation, the collected samples were brought directly to the laboratory in dark bottles.

2.2 Reagents

Standards used for chromatographic analyses were purchased from Sigma-Aldrich Co (St. Louis, MO, USA). Other reagents were of analytical grade and purchased from a Professional Lab (Casablanca, Morocco).

2.3 Accelerated storage test

Aliquots consisting of 30 mL from each sample were poured into glass vessels and kept closed in an oven (Binder

GmbH Bergstr.14 D-78532 Tuttlingen) at 60 °C (±1 °C). The samples were taken out of the oven at regular weekly intervals. The process of oxidation in progress was monitored by immediate measurement of the peroxide value (PV), UV extinction coefficients (K232 and K270), oil stability index (OSI) and free acidity.

2.4 Basic quality indices

Routinely quality indices were measured. These include free acidity (FA), expressed as % of oleic acid in the mass percentage of oil (g/100 g), peroxide value (PV) and given as milliequivalents of active oxygen per kilogram of oil (mEq O_2/kg oil). UV absorption coefficients at $\lambda = 232$ (K232) and $\lambda = 270$ nm (K270) as well as ΔK were determined. Saponification value (SV) is given as mg KOH/g (the number of milligrams of KOH needed to neutralize the fatty acids obtained by complete hydrolysis of 1 g of a given oil sample). To determine moisture content, 10 g of oil were placed in a ventilated oven at 103 °C at least for one hour until reaching a constant weight and then weighted after cooling. MC was expressed as percentage of weight loss. These quality indices were assessed following the analytical methods described by the standards [ISO 660:2020], [ISO 3960:2017], [ISO 6885:2016], and [ISO 3656:2002] and [ISO 3657:2020].

2.5 Fatty acids composition

Fatty acids were converted into their corresponding fatty acid methyl esters (FAME) through transmethylation according to the standard [ISO 12966-2:2017]. Ea, 0.1 g of each oil sample was sampled into a 10 mL screw-top test tube. Subsequently, 2 mL of isooctane was added and agitated. Then 0.1 mL of methanolic potassium hydroxide solution (2N) was added, instantly put on the cup and stirred for 1 minute. The solution was allowed to stand for 2 minutes. The solution gets clear and becomes cloudy again after a short time as the glycerol separates. After that, 2 mL of sodium chloride solution was added and agitated. The isooctane layer was extracted and transferred into a sample vial. Then about 1 g of sodium hydrogen sulfate was added and agitated. Fatty acid composition was investigated using a gas chromatography (Agilent-6890) coupled to a flame ionization detector (GC/FID). Capillary column CP-Wax 52CB (30 m × 250 μm i.d., 0.25 μm film thickness) was used. Helium (with a flow rate of 1 mL/min) was used as a carrier gas. The temperatures of the oven, injector, and detector were set at 185, 200, and 230 °C, respectively. The injection volume of the samples was 1 μL in a split mode (split ratio 1:50) as described in Ibourki *et al.* (2021).

The iodine value (IV) was computed from unsaturated fatty acids percentages using the formula: IV = (%C16:1 × 1.001) + (%C18:1 × 0.899) + (%C18:2 × 1.814) + (%C18:3 × 2.737) (Gharby *et al.*, 2020).

2.6 Sterols composition

The composition of the sterol fraction was measured according to ISO 12228-1:2014. In brief, 5 g of oil was saponified under reflux boiling during 1 h, by using 50 mL of ethanolic solution of potassium hydroxide (2 N). Subsequently, 100 mL of water was then added and the extraction of unsaponifiable matter was performed with 200 mL of hexane. Derivatives of the sterols were analyzed using an Agilent Technologies Varian 3800 A gas chromatography instrument equipped with a VF-1 ms (30 m and 0.25 mm i.d.). The temperature of the column was isothermal at 270 °C, the temperature of the injector and detector was 300 °C. The carrier gas was helium with 1.6 mL/min as a flow rate. Identification of individual peaks were carried out using available reference standards and comparing known retention times of the sterols in EVOOs. Three injections of 1 μL were applied for each sample. Results were expressed as mg of sterols per 100 g of oil (Seçilmis *et al.*, 2021; Oubannin *et al.*, 2022).

2.7 Tocopherols composition

Tocopherols content of the samples was performed as described in [ISO 9936:2016] method. The sample was prepared by dissolving olive oils in hexane, and injected into a normal phase HPLC employing a mixture of hexane 99.5% and isopropanol 0.5% as isocratic eluting system with a rate of flow of 1.0 mL/min. The injection volume was 10 μL for both sample and standard. Detection was administrated by means of a fluorescence detector (the fluorescence detector with $\lambda_{ex} = 280$ nm and $\lambda_{em} = 340$ nm) and quantification is obtained by means of external standardization with a mixture of single tocopherol forms preparing different calibration levels. All analyses were carried out in triplicate. Values were expressed as mean ± SD (Gharby *et al.*, 2021a, 2021b).

2.8 Oil stability index

The OSI of EVOOs was determined by the Rancimat method, which is considered an accelerated determination of oxidation. The Rancimat method evaluates oxidative stability by measuring the oxidation induction time, with the Rancimat apparatus (Metrohm 743, Herisau, Switzerland). 3 g of each sample were set at temperatures of 373, 383, 393, 403, 413, and 423 °K and at an airflow rate of 20 L/h were used to measure OSI (Farhoosh and Hoseini-Yazdi, 2014a; Gharby *et al.*, 2016a).

2.9 Kinetic data analysis

The temperature coefficient (T_C, °C^{-1}) was calculated using the slope (*a*-value) of the linear equation (1) between log OSI and temperature (T, °C).

$$\mathrm{Ln(k)} = \mathrm{a(T + b)}. \quad (1)$$

The values of the activation energy (Ea, kJ/mol) and the frequency factor (A, h^{-1}), were estimated using the slopes and intercepts from Arrhenius equation (2);

$$\mathrm{Ln(k)} = \mathrm{Ln(A)} + \left(\frac{\mathrm{Ea}}{\mathrm{RT}}\right), \quad (2)$$

Table 1. Mean values of Initial physicochemical parameters of evaluated EVOOs.

Quality indices	"Arbequina"	"Moroccan Picholine"	IOC
Free acidity (g/100 g)	0.80 ± 0.02	0.62 ± 0.01	<0.8
Peroxide value (mEq O_2/kg)	3.20 ± 0.51	2.10 ± 0.51	<20
Saponification value (mg KOH/g)	195.60 ± 0.11	194.50 ± 0.11	184–196
Iodine value (mg I_2/100 g)	85.90 ± 0.51	87.70 ± 1.03	75–94
K_{232}	2.10 ± 0.01	1.71 ± 0.01	<2.50
K_{270}	0.13 ± 0.01	0.16 ± 0.01	<0.22
ΔK	0.0009	0.002	<0.01
Moisture content (%)	0.04 ± 0.01	0.04 ± 0.01	0.2

IOC: International Olive Council. Results are expressed as mean ± SD ($n = 3$).

where, k is the reaction rate constant (the inverse of OSI), and R is the molar gas constant (8.3143 J/mol K).

The temperature acceleration factor (Q_{10}), was obtained from the equation (3);

$$Q_{10} = e^{-10Tc}. \quad (3)$$

Enthalpies (ΔH^{++}) and entropies (ΔS^{++}) of activation were obtained by regressing ln (k/T) *versus* (1/T) (K^{-1}) *via* the equation derived from the activated complex theory (eq. (4)). From the slopes and intercepts of the lines, ΔH and ΔS were calculated:

$$\mathrm{Ln}\left(\frac{k}{T}\right) = \mathrm{Ln}\left(\frac{kb}{h}\right) + \left(\frac{\Delta S}{R}\right) - \left(\frac{\Delta H}{RT}\right), \quad (4)$$

where kb is the Boltzmann constant ($1.380658 \times 10^{-}23$ J/K) and h is the Planck's constant ($6.6260755 \times 10^{-34}$ Js). The positive sign of ΔH^{++} reflects the endothermic nature of the formation of activated complex, showing that the reaction rate increases with temperature (Ramos *et al.*, 2020).

2.10 Statistical analysis

All determinations and measurements were carried out in triplicate. Statistical analysis (mean, standard deviation) was performed by using the Excel standard software package. The shelf lives of the samples were predicted by extrapolation of the graphs automatically drawn by the Origin line software according to the Rancimat method.

3 Results and discussion

3.1 Quality indices of studied EVOOs

The quality of the olive oils was studied by measuring parameters used regularly to measure the physical and chemical properties such as free acidity (FA), peroxide value (PV), specific coefficients of extinction at 232 and 270 nm (K232 and K270), and ΔK.

The quantification of free acidity (FA) in olive oils is highly relevant to their categorization and price (Dankowska and Kowalewski, 2019). The level of free fatty acids acts as a marker of TAG hydrolysis (Bijla *et al.*, 2021), which is strongly depending on the quality and freshness of olives used for the final product (Gazeli *et al.*, 2020). Mean values of acidity of both cultivars were 0.62 ± 0.01 g oleic acid (OA)/100 g and 0.80 ± 0.02 g oleic acid (OA)/100 g (Tab. 1). "Moroccan Picholine" recorded the lower acidity value, while "Arbequina" had the highest value. According to IOC standards (IOC, 2021), EVOOs from both cultivars seem to be classifiable as "extra virgin oils" as their free fatty acid content does not exceed 0.8 g oleic acid (OA)/100 g. Extra virgin olive oils are conceived as the best commercial quality olive oil since it is the healthiest and most sought out among all categories of olive oil as described by Dankowska and Kowalewski (2019). Similar behavior were previously described in some European olive cultivars (Dabbou *et al.*, 2009; Vekiari *et al.*, 2010). According to Rallo *et al.* (2018), free acidity, which is a quality criterion for olive oils, can show significant variations depending on genotype (cultivar), location and olive ripening among others (Rallo *et al.*, 2018).

Peroxide value (PV) provides a highly practical and satisfactorily sensitive criterion for assessing the initial oxidation state of vegetable oils (Gharby and Charrouf, 2021). The initial values found in this study were almost similar (Tab. 1) and they were in the order of 2.10 ± 0.51 mEq (O_2)/kg ("Moroccan Picholine") and 3.20 ± 0.51 mEq (O_2)/kg ("Arbequina"). In general, these values were relatively low, all below the limit recommended by the standards (20 mEq (O_2)/kg) (IOC, 2021). Our results are in accordance with other previously reported by Allalout *et al.* (2009), concerning four EVOOs from super intensive Spanish and Greek cultivars grown in northern Tunisia. Nevertheless, our results are below those of Moroccan and Turkish olive oils reported by Zaroual *et al.* (2021) and Demirag and Konuskan (2021).

K232 and K270 values are typically another measure of the oxidative state of oils quality (El Moudden *et al.*, 2020), which measure conjugated dienes and trienes and their secondary oxidation products (Pardo *et al.*, 2021). As shown in Table 1, both "Arbequina" and "Moroccan Picholine" cultivars showed low values within the range of 1.71 ± 0.011 to 2.10 ± 0.01 (K232) and from 0.13 ± 0.01 to 0.16 ± 0.01 (K270), respectively. ΔK values of oil samples were found to be ΔK = 0.0009 ("Arbequina") and ΔK = −0.002 ("Moroccan Picholine"). All analyzed oils exhibited K232, K270, and ΔK values within the standards established (IOC, 2021). The spectrophotometric values were found to be in the range of previously published values for olive cultivars, covering seven olive-growing areas in northern Morocco (Bajoub *et al.*, 2015).

Iodine value (IV) highlights the degree of unsaturation of oils, overall number of double bonds present in fats and oils (Obimakinde *et al.*, 2015; Bijla *et al.*, 2021). As shown in

Table 2. Authorized values (IOC) and initial fatty acid, sterol, and tocopherol composition of evaluated EVOOs.

Fatty acids (g/100 g)	IOC (2021)	"Arbequina"	"Moroccan Picholine"
Palmitic ac.	7.5–20	14.30 ± 1.51	9.21 ± 1.51
Stearic ac.	0.5–5	2.01 ± 0.51	2.90 ± 0.51
Oleic ac.	55–83	67.10 ± 2.51	74.60 ± 2.51
Linoleic ac.	3.5–21	13.20 ± 1.51	10.70 ± 1.51
Linolenic ac.	<1	0.80 ± 0.11	0.90 ± 0.11
Sterols (g/100 g)			
Cholesterol	<0.5	0.10 ± 0.01	0.10 ± 0.01
Brassicasterol	<0.1	0.10 ± 0.01	0.10 ± 0.01
Campesterol	<4	2.70 ± 0.20	3.10 ± 0.31
Stigmasterol	<Campesterol	1.70 ± 0.10	1.90 ± 0.21
Other sterols	>93	93.80 ± 80.01	94.80 ± 8.02
Total sterols	>100	241.20 ± 10.01	207.00 ± 10.01
Tocopherols (mg/kg)			
α-Tocopherol		166.30 ± 15.02	167.01 ± 15.01
β-Tocopherol		11.70 ± 3.11	10.50 ± 2.51
γ-Tocopherol		1.90 ± 0.31	2.30 ± 0.31
δ-Tocopherol		21.90 ± 6.01	20.10 ± 6.11
Total	150–250	202.00 ± 21.01	182.00 ± 30.01

Results are expressed as mean ± SD ($n = 3$)

Table 1, the observed values were quite similar, "Moroccan Picholine" was found to have a high value (87.70 ± 1.03 mg I_2/100 g) compared to "Arbequina" (85.90 ± 0.51 mg I_2/100 g). The iodine value depends directly on the number of double bonds present in oil (Afzal *et al.*, 2021). In general, the iodine values were satisfactory and equivalent to international standard of olive oil (75–94 (I_2)/100 g) (IOC, 2021). Our results were found to be in the range of previously published literature (Borchani *et al.*, 2010; Selka *et al.*, 2019).

Saponification value (SV) is a measure of the average molecular weight of the triglycerides present in oils (Bijla *et al.*, 2021). The SV correlates directly with the average molecular weight of lipids. The lower the average molecular weight, the higher the SV (Afzal *et al.*, 2021). Table 1 represents the comparison among the samples regarding the saponification value. The observed values were quite similar; 194.50 ± 0.11 mg KOH/g ("Moroccan Picholine") and 195.60 ± 0.11 mg KOH/g ("Arbequina"). These values are within the limits of the saponification average established by the international standard of olive oil (IOC, 2021). Our results are almost comparable to the saponification values of Pakistani and Algerian olive oils reported by Selka *et al.* (2019) and Afzal *et al.* (2021).The International Olive Oil Council requires a moisture and volatile matter levels of less than 0.2 for extra virgin and virgin quality olive oils, respectively. Referring to this regulation, both samples showed a similar value 0.04 ± 0.01%, below the IOC allowable limits and moisture and volatile matter levels was found to be in the range of previously published by (Karunathilaka *et al.*, 2020).

3.2 Fatty acids composition

Vegetable oils are typically found in nature as triglycerides, which are esters of glycerol and fatty acids. These seem to establish their nutritional behavior (Wabaidur *et al.*, 2016; Ibourki *et al.*, 2022), and largely affects their viscosity and sets their melting point. In addition, these compounds significantly influence the chemical and nutritional properties of the olive oil (Lukić *et al.*, 2021). Our results for fatty acids are represented in Table 2.

Interestingly, the results showed that both cultivars stand out by their high content in monounsaturated fatty acids (MUFA). The results showed a fairly wide range of values and that both oils are excellent source of oleic acid (C18:1), which was the most predominant fatty acid in range of 67.10% ("Arbequina") and increased up to 74.60% in "Moroccan Picholine". Palmitic acid (C16:0) constituted the second most notable amount in "Arbequina" (up to 14.30%), meanwhile the linoleic acid (C18:2) was in the third place with 13.20%. The results obtained in "Arbequina" oil were in agreement with those found by other authors (Borges *et al.*, 2017). Contrary to the "Moroccan Picholine", linoleic acid (C18:2) was the second most remarkable acid (up to 10.70%) while the palmitic acid (C16:0) was the third with 9.21%. Linolenic acid (C18:3), is a highly oxidizable molecule (Gharby and Charrouf, 2021; Ibourki *et al.*, 2022), displayed the lower acid content in both samples (0.80–0.90%). However, this small content of linolenic acid can be used to detect the adulteration of olive oil with other vegetable oils rich in linolenic acids such as rapeseed (up to of 13%). and soybean (up to of 11%) (Sakar and Gharby, 2022). The other fatty acids such as myristic acid (C14:0), palmitoleic acid (C16:1), arachidic acid (C20:0), and behenic acid (C22:0) were found only in relatively lower quantities <1%. The results obtained were in agreement with the results previously reported by other authors (Afzal *et al.*, 2021; Shen *et al.*, 2021). According to Pardo *et al.* (2021), the fatty acid composition occasionally might show a quite wide range of values in virgin olive oils, owing to genetic and environmental factors during fruit development, as well as the stage of ripening of the olives at the time of harvest.

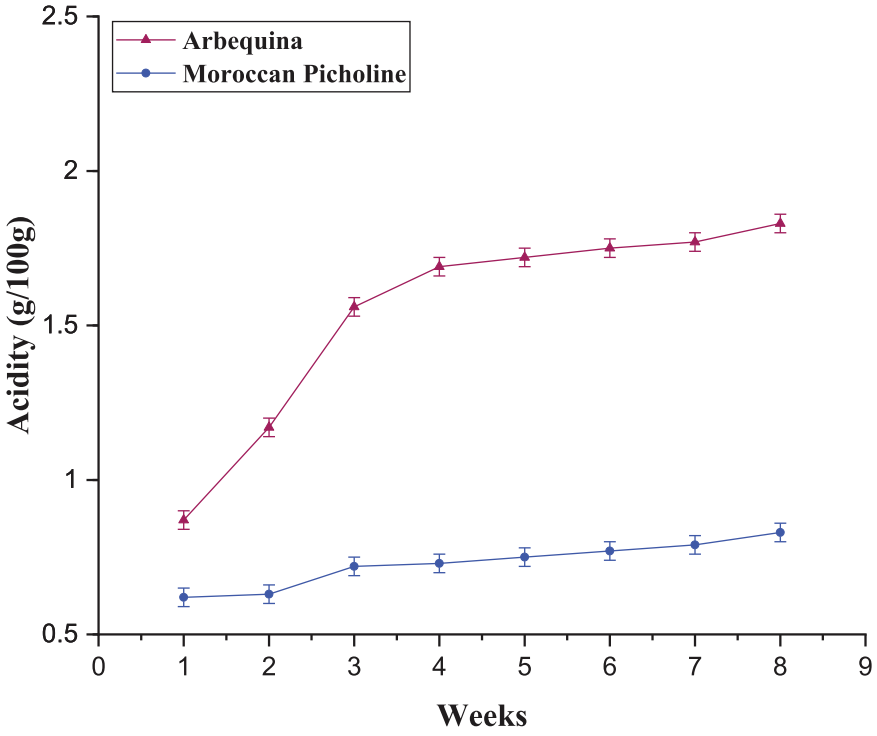


Fig. 1. Evolution of free acidity in EVOOs subjected to accelerated storage conditions at 60 °C. Results are expressed as mean ± SD ($n = 3$).

3.3 Sterols composition

Sterols constitute an important fraction of the unsaponifiable part of olive oil (Gharby *et al.*, 2018). Despite representing only a minor part, however they are known by their chemical characteristics and especially their antioxidant properties (Gagour *et al.*, 2022). They also bring a nutritional value and determine the organoleptic properties of olive oil (Gharby *et al.*, 2021a, 2021b). In addition, the use of sterol content profiles have been proposed for classifying virgin olive oils in terms of their variety, making their determination an important tool to identify the authenticity of olive oil and detect adulterations (Lukić *et al.*, 2021). As shown in Table 2, levels of sterol profiles were quite similar in both cultivars. The highest level recorded was in β-sitosterol, being the main sterol in the studied olive oils (93.80–94.80%). The other sterols were minor such as stigmasterol, cholesterol, and brassicasterol (Tab. 2). EVOOs from both cultivars showed a phytosterol composition in compliance within the established limits (IOC, 2021). In contrast to our data, a number of authors have observed a significant difference in the amount of sterols between different cultivars, stating that this type of compound depends mainly on the geographical area and the effects of irrigation of olive oils (Mansouri *et al.*, 2015; Mikrou *et al.*, 2020).

3.4 Tocopherols composition

Besides unsaturated fatty acids and sterols, tocopherols are important carriers of bioactive properties of olive oil, and are partly responsible for its beneficial effects of its consumption (Gharby *et al.*, 2016b; Lukić *et al.*, 2021). Tocopherols also contribute to the preservation of oils by serving as natural preservatives against autoxidation (Harhar *et al.*, 2014). As shown in Table 2, among these isomers of tocopherol, α-tocopherol, which had the highest vitamin E activity and several nutritional benefits, was the most predominant tocopherol detected in both oil cultivars (166.30 ± 15.02–167.00 ± 15.01 mg/kg), representing over 90% of their total content. The observed values of tocopherol content in our olive cultivars were similar to that found in olive oil cultivars (158.6 mg/kg) grown in Spain (Jenisová *et al.*, 2021). High tocopherol level than that found by other authors (116 mg/kg, Mikrou *et al.*, 2020), regarding olive oils grown in Greece. Wang *et al.* (2021) found a significant difference between three Chinese cultivars, ranging from 157.86 to 440.13 mg/kg. According to Mikrou *et al.* (2020), several factors may significantly influence the amount of minor compounds of olive oil, among them genetic and agronomic factors (harvest date and year of cultivation).

3.5 Oxidative stability during storage

3.5.1 Changes in free acidity

Such quality parameters as the specific extinctions at 232 and 270 nm, free acidity, and peroxide value were followed up in order to study the effect of storage in accelerated conditions (oven test at 60 °C) on the studied EVOOs.

As a consequence of the accelerated storage, free acidity showed a gradual increase for both cultivars (Fig. 1). Likewise, a drastic and much faster increase was observed in "Arbequina" oil in the first 3 weeks of storage ranging from 0.80 ± 0.02 to 1.56 ± 0.01 g oleic acid (OA)/100 g (an increase

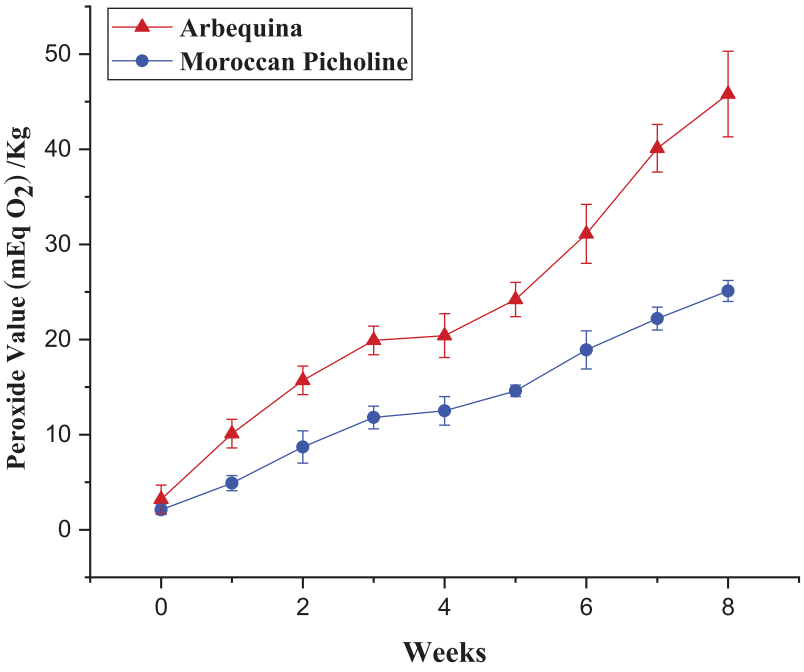


Fig. 2. Evolution of peroxide value (PV) of EVOOs subjected to accelerated storage conditions at 60 °C.

of 95%), which lost the "extra virgin" label only after 1 week of storage (0.87 ± 0.01 g oleic acid (OA)/100 g). Unlike "Moroccan Picholine" which varied from 0.62 g/100 g to 0.72 g oleic acid (OA)/100 g (+16%), in 1–3 weeks. After this period, the progression increased slowly and in parallel until 3–8 weeks at a similar rate. The highest levels were observed in "Arbequina" oil (1.83 g/100 g after 8 weeks of storage), compared to "Moroccan Picholine" which therefore possessed the lowest levels of free acidity until the end of the storage period (0.83 g oleic acid (OA)/100 g after 8 weeks).

The increase in FA at higher temperatures (60 °C) could be due to increased hydrolytic activity of lipase on triacylglycerols remaining in the oil during accelerated storage (Grossi *et al.*, 2019). Therefore, both oils lose the label of extra virgin oils falling into the category "virgin oils" following the International Olive Oil Council. Similar results were obtained in previous studies showing an increase in FA level, along with storage at room temperature for olive oil (Stefanoudaki *et al.*, 2010; Mousavi *et al.*, 2021), and for argan oil (Gharby *et al.*, 2021a, 2021b; Oubannin *et al.*, 2022). However, based on the data found by Shendi *et al.* (2019) and Caipo *et al.* (2021), FA values of Turkish and Chilean extra virgin olive oils, remain below the limit established by the International Olive Council standards, during the whole storage period (12 months) at different conditions.

3.5.2 Changes in saponification value

Saponification value plays an important role in quality control and identification of lipids (Borchani *et al.*, 2010). It is a measure of oxidation during storage and also indicates the deterioration of oils (Ibeto *et al.*, 2012). As a consequence of the accelerated storage, a slight decrease of this parameter was observed to a similar degree in both investigated samples dropping from 195.65 ± 0.08 mg KOH/g oil to 193.07 ± 0.08 mg KOH/g oil for "Arbequina", and from 194.49 ± 0.07 mg KOH/g oil to 180.75 ± 0.03 mg KOH/g oil for "Moroccan Picholine", which had the lowest saponification values in different storage periods. In the same line with our results, other authors (Abdalla *et al.*, 2014), reported a decrease in saponification values in 10 samples of EVOOs collected from cooperatives for olive growers in northern Morocco. Our results were also in concordance with the result previously reported by Méndez and Falqué (2007), during 6 months of different storage conditions.

3.5.3 Changes in peroxide value

Peroxide value is a reliable parameter to assess the peroxide content and indicates the degree of oxidation of an oil in the early stages of oxidative rancidity (Fadda *et al.*, 2022). Then it is a very practical criterion with satisfactory sensitivity to appreciate the early stages of oxidative deterioration during storage (Gharby *et al.*, 2016b).

Our results highlighted that, the PV of both cultivars increased significantly, and the acceleration is accentuated in parallel during the storage period (1–6 weeks) at 60 °C. Thus, a strong and extended evolution was observed in "Arbequina" throughout the storage period (6–8 weeks), reaching a higher value (45.80 ± 4.50 mEq (O_2)/kg), an increase 6 times higher than the initial state. This evolution was twice as great compared to the evolution of "Moroccan Picholine", which was characterized by a slower dynamic ranging from 4.9 ± 0.8 to 25.1 ± 1.10 mEq (O_2)/kg at the end of the assay.

Nevertheless, the increase in PV at higher temperatures (Fig. 2), in both cultivars exceeding the legal limits (20 mEq (O_2)/kg) suggested that oxidation of susceptible components of olive oil could take place under accelerated

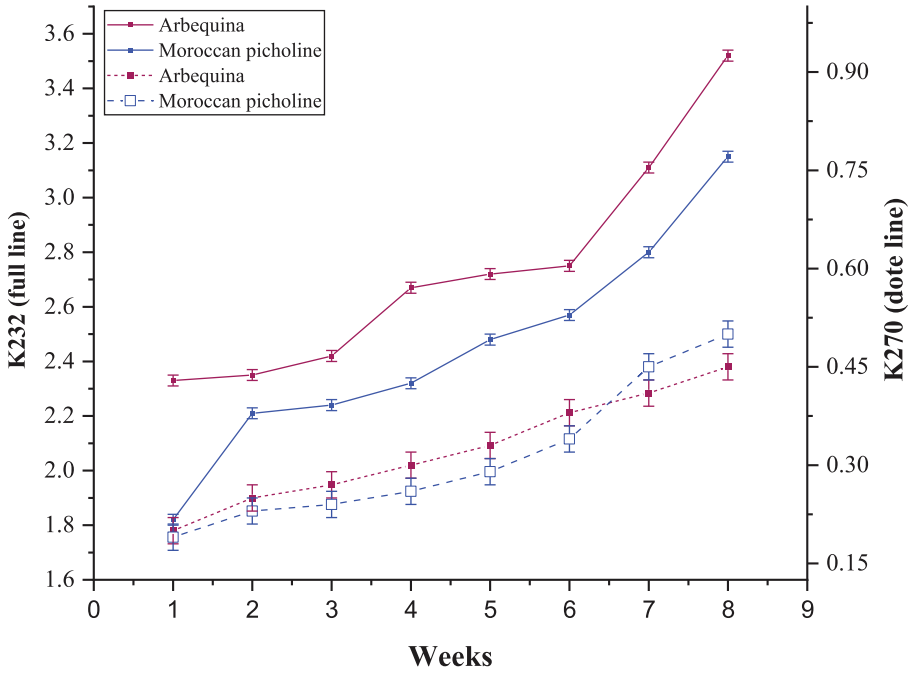


Fig. 3. Evolution of conjugated dienes (K232) and triene K270 of EVOOs subjected to accelerated storage conditions at 60 °C.

conditions during storage (Sakar and Gharby, 2022). This behavior could be explained by the decomposition of hydroperoxides and subsequent formation of oxidation secondary products. The decrease in stability of 'Arbequina' compared to 'Moroccan Picholine' might be caused by the reduced content of minor compounds such as phenols and tocopherols that prevent the formation of peroxides during accelerated storage. Similar findings were reported in previous studies, showing an increase in PV upon different storage periods (Stefanoudaki *et al.*, 2010; Farhoosh and Hoseini-Yazdi, 2014a; Mousavi *et al.*, 2021).

3.5.4 Changes in K232 and K270

K232 indicates that the primary oxidation products were formed more rapidly in "Arbequina" than "Moroccan Picholine" during the whole storage period (8 weeks, Fig. 3), underwing a sharp and strong increase ranging from 2.33 ± 0.01 to 3.52 ± 0.01 in "Arbequina". Same trend was observed in "Moroccan Picholine" which showed very low levels (1.82 ± 0.01 to 3.15 ± 0.01).

K270 increased during storage period in both cultivars. As expected, a much faster increase was observed in "Arbequina" compared to "Moroccan Picholine" (Fig. 3), which allowed maintaining very low levels until the sixth week (1–6 weeks). However, afterwards, a remarkable increase was detected in the later surpassing the evolution of "Arbequina" during the following 2 weeks (6–8 weeks). The obtained values explain that the formation of secondary oxidation products has already begun, and these values remained out of the limit for EVOO category considering that the maximum permitted values of K232 and K270 for EVOOs label were 2.50 and 0.20, respectively.

Abbadi *et al.* (2014) studied the effect of packaging materials and storage temperatures on quality degradation of EVOOs from olives grown in Palestine. According to these authors, the increase in K232 and K270 values, occurs very frequently between the extraction of olive oil and its consumption depending on the storage time and conditions (Abbadi *et al.*, 2014). As our results, other authors reported an increase in K232 and K270 in four cultivars of Moroccan olive during 12 months of storage (Essiari *et al.*, 2015). However, Di Stefano and Melilli (2020) reported that after 12 months, the stored Italian olive oils showed an evolution of K232 for all oils ranging from 1.90 to 2.21 without exceeding the limit (2.5) defined by the standards (IOC, 2021). Initial values of K270 were about 0.06–0.11 and the final values at the end of the experiment were 0.12–0.19 and then lower than their threshold value (Di Stefano and Melilli, 2020).

3.5.5 Changes in fatty acids composition

Fatty acid composition is an important measure of quality as the proportions of individual fatty acids determine the physical properties and nutritional value of the oil (Gharby *et al.*, 2021a, 2021b). The studied storage conditions did not appear to have any effect on fatty acid composition as shown in Table 3. The data showed a slight decrease in the amount of polyunsaturated fatty acids (C18:2) at the end of storage period (8 weeks), whereas the amount of saturated fatty acids (C16:0–C18:0) increased slightly, but these were not statistically significant in both oils. Similar findings were observed by Gargouri *et al.* (2015), which confirmed the stability of the fatty acid composition during 6 months of storage.

Table 3. Evolution of fatty acids (g/100 g) of EVOOs subjected to accelerated storage conditions at 60 °C.

Storage time (weeks)	Fatty acids	"Arbequina"	"Moroccan Picholine"
0	C16:0	14.30 ± 1.50	9.20 ± 1.51
	C18:0	2.00 ± 0.50	2.90 ± 0.50
	C18:1	67.10 ± 2.50	74.60 ± 2.51
	C18:2	13.20 ± 1.50	10.70 ± 1.50
	C18:3	0.80 ± 0.10	0.90 ± 0.10
3	C16:0	14.40 ± 1.50	9.20 ± 1.01
	C18:0	2.01 ± 0.52	2.90 ± 0.40
	C18:1	67.22 ± 12.01	74.80 ± 12.01
	C18:2	13.10 ± 5.01	10.70 ± 5.01
	C18:3	0.70 ± 0.20	0.90 ± 0.31
6	C16:0	15.00 ± 5.01	9.60 ± 1.50
	C18:0	2.00 ± 0.50	2.90 ± 0.50
	C18:1	67.30 ± 10.50	74.60 ± 10.50
	C18:2	12.70 ± 5.01	10.40 ± 5.01
	C18:3	0.60 ± 0.20	0.90 ± 0.10
8	C16:0	15.90 ± 5.01	9.80 ± 3.00
	C18:0	2.10 ± 0.50	2.90 ± 0.50
	C18:1	68.70 ± 10.01	74.90 ± 15.01
	C18:2	10.20 ± 1.01	10.20 ± 1.01
	C18:3	0.30 ± 0.10	0.70 ± 0.10

Results are expressed as mean ± SD ($n = 3$).

Table 4. Evolution of tocopherols content (mg/100 g) of EVOO subjected to accelerated storage conditions at 60 °C.

Storage time (weeks)	Tocopherol	"Arbequina"	"Moroccan Picholine"
0	α-T	166.30 ± 15.01	167.00 ± 15.01
	β-T	11.70 ± 3.01	10.50 ± 2.50
	γ-T	1.90 ± 0.30	2.30 ± 0.30
	δ-T	21.90 ± 6.01	20.10 ± 6.01
	Total	202.00 ± 21.01	182.00 ± 30.01
3	α-T	125.30 ± 15.01	129.80 ± 18.01
	β-T	7.50 ± 1.50	9.10 ± 1.01
	γ-T	1.90 ± 0.50	2.05 ± 0.50
	δ-T	9.30 ± 2.00	1.90 ± 0.50
	Total	144.00 ± 21.01	143.30 ± 19.01
6	α-T	95.10 ± 15.01	91.60 ± 10.01
	β-T	5.80 ±.15.01	9.10 ± 1.01
	γ-T	2.30 ± 0.50	2.00 ± 0.50
	δ-T	15.90 ± 6.01	1.30 ± 0.20
	Total	119.40 ± 45.01	104.80 ± 15.00
8	α-T	60.00 ± 10.01	82.20 ± 11.01
	β-T	4.10 ± 1.50	8.60 ± 1.01
	γ-T	1.30 ± 0.50	2.00 ± 0.21
	δ-T	9.80 ± 1.01	0.50 ± 0.10
	Total	75.50 ± 12.01	94.30 ± 15.00

Results are expressed as mean ± SD ($n = 3$).

3.5.6 Changes in tocopherols composition

The measurement of tocopherols content during the accelerated storage is very important, owing to their protective role against oxidative detection in oils (Harhar *et al.*, 2014). Table 4 shows changes in total tocopherols content of the studied EVOOs as a function of storage time and temperature. In parallel with stability, the total tocopherols content reduced dramatically along with α-Tocopherol over the time regardless of the storage temperature in both investigated samples (Tab. 4). As expected, "Arbequina" showed a sharp reduction in tocopherol content compared to "Moroccan Picholine". For instance, the total tocopherol amount of "Arbequina" decreased from 202.00 ± 21.01 to 75.50 ± 12.01 mg/kg (fell by 62%) after 8 weeks of storage at 60 °C, while "Moroccan Picholine" dropped from 182.00 ± 30.01 to 94.30 ± 15.00 mg/kg (fell by 48%). In general, the marked decrease in the tocopherols content reflects that this natural antioxidant was first destroyed during the oxidative process supporting the findings of Shendi *et al.* (2018). Many reports recorded also significant losses in these components after storage. For instance, Shendi *et al.* (2018) and Shendi *et al.* (2020) reported important losses in the tocopherols compounds of EVOOs after 12 and 24 months of storage at room temperature, respectively. The tocopherols are recognized as powerful lipid radical scavengers and a strong antioxidant capacity, unfortunately, these natural substances are highly dependent on various parameters such as high temperature, high oxygen availability, level of polyunsaturated fatty acids, and their composition, as well as the presence of prooxidants such as metal ions, heavy metals among others (Sakar and Gharby, 2022). As the oils lose their oxidative stability during the oxidation process (Tab. 4), it can be anticipated that the antioxidative system is being slowly depleted.

3.6 Oil stability index

Oxidative stability of oils under accelerated conditions is typically estimated by the Rancimat method (Mancebo-Campos *et al.*, 2007). Rancimat test indicates the resistance to the oxidation process of the product characterized by radical reactions (Nieto *et al.*, 2010). The induction times of the olive oil samples at 100–150 °C are presented in Table 5. Considering the oil stability index (OSI) at 100 °C, the differences in both kinds of olive oils were significant. For example, "Moroccan Picholine" showed higher oxidative stability (OSI = 43.6 h at 100 °C) *versus* "Arbequina", which showed merely 25.10 ± 1.00 h under the same conditions. As can be seen in Table 5, the OSI decreased significantly in a linear pattern with increasing temperature in both cultivars, revealing the greater stability for "Moroccan Picholine" at 120–130–140–150 °C. The resistance to oxidative deterioration according to Nieto *et al.* (2010), is generally assigned to two major factors; the first, the composition of fatty acids, which in the case of our samples "Moroccan Picholine" (has a high ratio MUFA/PUFA). The second, the level of minor compound pool of antioxidant activity, which in this case consists mainly of tocopherols and polyphenols but also chlorophylls and carotenoids (Nieto *et al.*, 2010). Our values were similar to those reported for EVOOs (Ciemniewska-Zytkiewicz *et al.*, 2014; Farhoosh and Hoseini-Yazdi, 2014a).

Table 5. Induction time and the reaction rate constants k of the evaluated EVOOs.

	Induction time (h)					
Temperature [°K]	373	383	393	403	413	423
"Arbequina"	25.10 ± 1.00	11.90 ± 1.50	5.90 ± 1.00	3.10 ± 1.00	1.40 ± 0.50	0.60 ± 0.50
"Moroccan Picholine"	43.60 ± 1.01	21.30 ± 1.01	9.70 ± 1.50	4.80 ± 1.01	1.90 ± 10.01	1.10 ± 0.50
	Reaction rate constant k ($\times 10^{-3} h^{-1}$)					
"Arbequina"	39.84	84.03	169.49	322.58	714.29	1666.67
"Moroccan Picholine"	22.94	46.95	111.11	208.33	526.32	909.09

3.7 Kinetic parameters of oxidative reaction

Since oxidation of edible oils is the most important reaction that causes deterioration of oil quality, kinetic analyses were attempted to examine the deterioration behavior of virgin olive oils, and its effect on the shelf life from a quantitative standpoint. As a result, the determination of the parameters of the kinetic model and the Arrhenius relationship that best fit the experimental data was performed.

3.7.1 Reaction rate constants of olive oils at temperatures (373–423 °K)

The k values for lipid oxidation of each sample, at each temperature are presented in Table 5. When examining the rates of lipid oxidation as a function of temperature, the results showed a marked effect of temperature on the oxidative behavior in both cultivars. It can be observed that the rate increases as the temperature increases in parallel and consecutive manner. Our results are in agreement with those found by other authors relating to olive oils of Moroccan origin (Gharby *et al.*, 2021a, 2021b).

3.7.2 Kinetic and thermodynamic parameters of olive oils

As shown in Table 6, the semi-logarithmic relationship between k and T values (Ln (k) = a (T) + b), for the samples ranged from 0.0751 K^{-1} ("Moroccan Picholine") to 0.0735 K^{-1} ("Arbequina"), which were in agreement with the findings outlined recently (Farhoosh *et al.*, 2008; Gharby *et al.*, 2021a, 2021b). As shown in Figure 4, the semi-logarithmic relationship between k and T values in both cultivars demonstrated a linear dependency with a strong correlation of determination ($R^2 = 0.99$). The T_{Coeff} values of the olive oils studied in this work were also in accordance with the literature data or slightly higher (in absolute value) (Farhoosh and Hoseini-Yazdi, 2014b; Veloso *et al.*, 2020). T_{Coeff} values are highly influenced by the quality grade and chemical composition of oil (*e.g.* individual fatty acids and phenolic compositions), cultivar, or even the individual phenolic compositions for the same cultivar (Harhar *et al.*, 2018; Veloso *et al.*, 2020).

3.7.3 Arrhenius equation parameters

In order to investigate the formation of secondary oxidation products under Rancimat conditions, we determined the regression parameters of the Arrhenius relations between the reaction rate constant and the temperature for the two studied olive oils ("Arbequina" and "Moroccan Picholine"). The frequency factors (A (h^{-1})), activation energies (Ea (kJ/mol)) and numbers (Q_{10}) were also determined (Tab. 6).

By using Ea (activation energy), which is the minimum amount of requited energy for a chemical reaction to take place (Farhoosh and Hoseini-Yazdi, 2014b). Ea of "Moroccan Picholine" oil (98.44 kJ mol^{-1}) was slightly higher compared to "Arbequina" oil (96.28 kJ mol^{-1}). As the formation of the first free radical that initiates the autoxidation reaction requires a considerable amount of energy (Jaimez-Ordaz *et al.*, 2019). The results showed that "Arbequina" was more susceptible to deterioration under Rancimat conditions, while "Moroccan Picholine" was more stable under the same conditions. According to Gharby *et al.* (2016a) and Yang *et al.* (2018), Ea value of a given vegetable oil is impacted by the ratio of unsaturated compounds. Once again, our results are quite similar to those reported by Heidarpour and Farhoosh (2018) for Iranian olive oils (Ea = 104.58 ± 0.97–105.05 ± 1.94 kJ mol^{-1}). Ea cannot be considered as a unit representative of the rate of lipid oxidation or the oxidative stability in the whole lipid systems which are of very high complexity (Farhoosh and Hoseini-Yazdi, 2014a; Mahdavianmehr *et al.*, 2016). Frequency factor (A) is an important complementary kinetic parameter that determines the rate of oxidation reaction of oils (Farhoosh and Hoseini-Yazdi, 2014b). Following the same trend as Ea, frequency factor (A), showed a little variation between the samples ranging from $1.12 \times 10^{15} h^{-1}$ ("Arbequina") to $1.34 \times 10^{15} h^{-1}$ ("Moroccan Picholine"). Correlation between the activation energy and the frequency factor (A) has been proven by other previous studies (Veloso *et al.*, 2020; Gharby *et al.*, 2021a, 2021b).

Following the activated complex theory, the entire oxidation reaction is started by the formation of an activated complex, whereby the reactive molecules rearrange their chemical structure and bonds closely to each other, causing the required intermolecular reactions to create the final products of lipid oxidation (Farhoosh and Hoseini-Yazdi, 2014b). The values of ΔH^{++} and ΔS^{++} are regarded to be the thermal energy needed for the steric changes and level of disorder, respectively, of the activated reactant molecules in the system (Mahdavianmehr *et al.*, 2016). The values of ΔH^{++} and ΔS^{++} of the activated complexes formed during the initiation peroxidation of "Arbequina" and "Moroccan Picholine" oils were estimated to be 92.35 kJ mol^{-1} *versus* 94.617 kJ/mol and 89.803 J/mol K *versus* 88.306 J/mol K, respectively. ΔH^{++} and ΔS^{++} had similar patterns as activation energies (Ea) and frequency factors (A) which is in agreement with the literature data for olive oils (Farhoosh and Hoseini-Yazdi, 2014b). While the values estimated in the present study (ΔH^{++}) were similar

Table 6. Arrhenius kinetic parameters, frequency factors, activation energy, temperature acceleration factor, activation enthalpies, and entropies for lipid oxidation of the evaluated EVOOs.

	"Arbequina"	"Moroccan Picholine"
Ln (k) = a (T) + b	Ln (k) = 0.0735 (T) − 16.851	Ln(k) = 0.0751 (T) − 17.956
a	0.0735	0.0751
b	−16.851	−17.956
R^2	0.9982	0.9969
T_{coef} x 10−2 (K^{-1})	7.31	7.51
Ln (k) = Ln (A) − (Ea/R) × (1/T)	Ln (k) = −11.511 × (1/T) + 41.36	Ln (k) = −11.774 × (1/T) + 41.538
a	−11.511	−11.774
b	41.36	41.538
R^2	0.995	0.997
A [h^{-1}]	$1.12016E^{+15}$	$1.34107E^{+15}$
Ea [kJ/mol]	96.28	98.44
Q_{10}	2.07	2.11
	Ln (k/T) = Ln (kB/h) + (S/R) − (H/R) × (1/T)	
a	−11.110	−11.380
b	34.381	34.561
R^2	0.994	0.996
ΔH^{++} [kJ/mol]	92.372	94.617
ΔS^{++} [J/mol K]	88.306	89.803

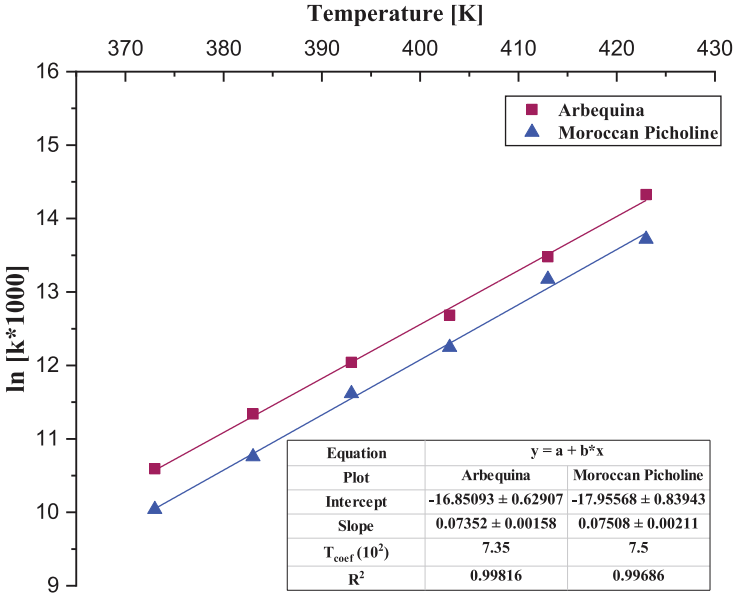
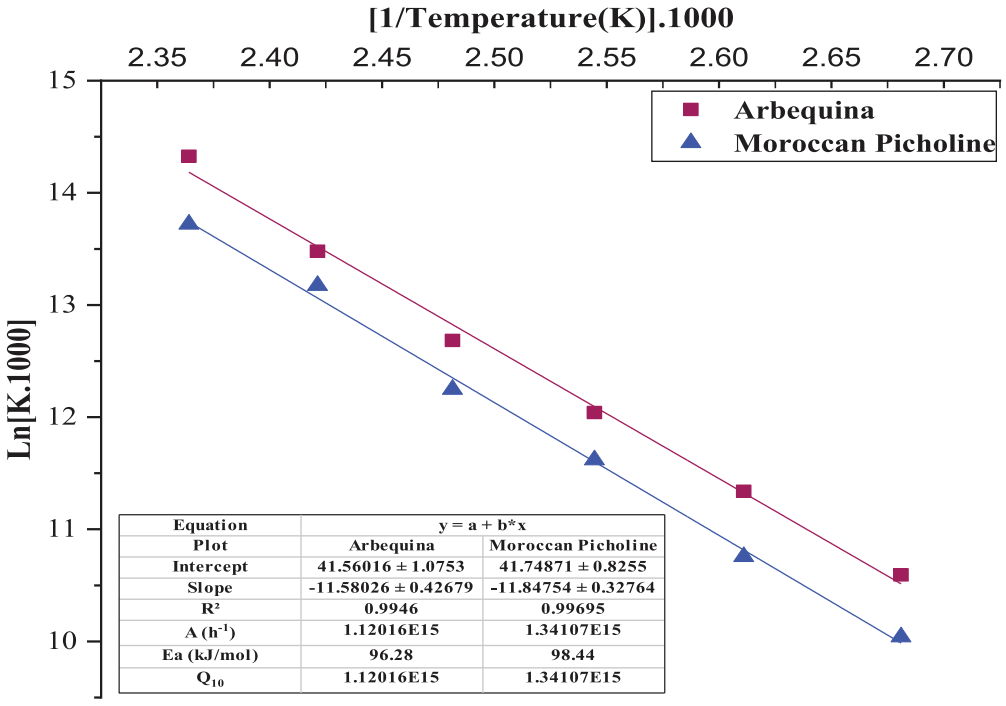


Fig. 4. Semi-logarithmic relationship between k and T values for lipid oxidation of the evaluated EVOOs.

to those found by Farhoosh *et al.* (2008) for olive oils (83.64 kJ mol^{-1}) and slightly greater than those found by Mahdavianmehr *et al.* (2016) for pure triacylglycerols extracted from olive oils (65.50 kJ mol^{-1}) (Figs. 5 and 6).

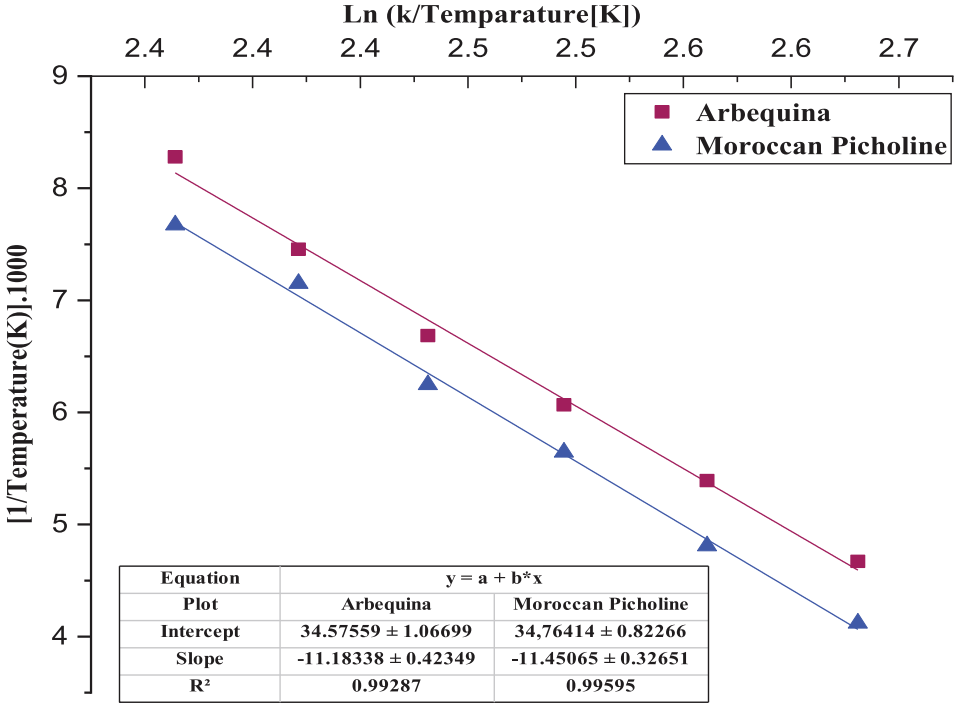
3.8 Shelf life prediction

Estimating expected shelf life of edible oils is therefore of great interest before their marketing (Kochhar and Henry, 2009). Shelf life was assessed using the accelerated Rancimat test method, which serves as an easy-to-implement test method to quickly monitor oxidative stability of oils and fats (Farhoosh, 2007). Induction time was measured at elevated temperatures (100, 110 °C...) within few hours and by plotting the logarithm of the times *versus* temperature, the shelf life of the oils can be estimated at room temperatures (25 °C) (Upadhyay and Mishra, 2015). The estimated shelf life of the samples at 25 °C by extrapolation is presented in Figure 7. At room temperature, "Arbequina" was found to have a shorter induction time of 9 months, implying that "Arbequina" will have a shorter shelf life compared to "Moroccan Picholine", with an anticipated time span of usability of 17 months when



Equation	y = a + b*x	
Plot	Arbequina	Moroccan Picholine
Intercept	41.56016 ± 1.0753	41.74871 ± 0.8255
Slope	-11.58026 ± 0.42679	-11.84754 ± 0.32764
R^2	0.9946	0.99695
A (h^{-1})	1.12016E15	1.34107E15
Ea (kJ/mol)	96.28	98.44
Q_{10}	1.12016E15	1.34107E15

Fig. 5. Semi-logarithmic relationship between the values (k/T) and (1/T) for the oxidation of lipids of the evaluated EVOOs.



Equation	y = a + b*x	
Plot	Arbequina	Moroccan Picholine
Intercept	34.57559 ± 1.06699	34,76414 ± 0.82266
Slope	-11.18338 ± 0.42349	-11.45065 ± 0.32651
R^2	0.99287	0.99595

Fig. 6. Semi-logarithmic relationship between the values (k/T) and (1/T) for the oxidation of the evaluated EVOOs.

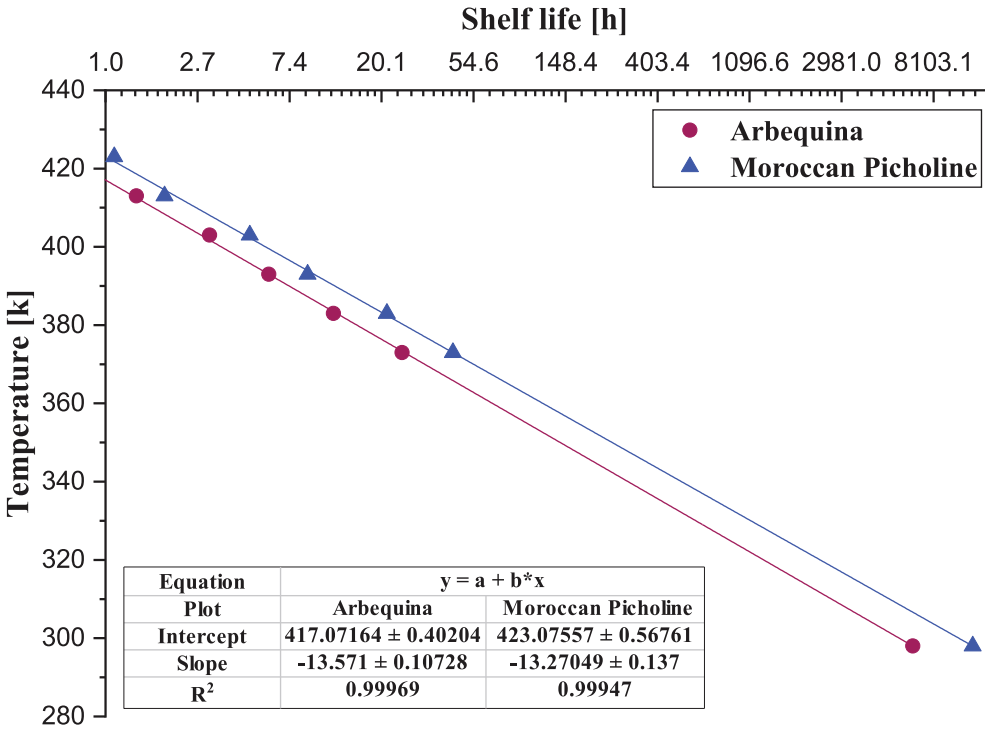


Fig. 7. Extrapolation at 25 °C (298 °K) of the shelf life of the evaluated EVOOs.

extrapolated to 25 °C. The shelf life ratings agree with the other outcomes of the oxidative state kinetic parameters and bioactive compounds, particularly the tocopherols profile, which showed a significant loss after the accelerated storage period.

4 Conclusion

In this paper, olive oil quality from two Mediterranean cultivars ("Arbequina" and "Moroccan Picholine") was assessed through a set of analytical parameters. Our study highlighted variations more or less considerable between both cultivars in terms of the studied parameters. Our comparative study shows also that accelerated storage had a significant effect on oils quality. In this study period (8 weeks), "Arbequina" was quite sensitive to oxidation compared to "Moroccan Picholine". This is evidenced by the increase of some important indicators of lipid alteration (AV, PV, K232, and K270), as well as by a slight increase of oleic acid percentage in the fatty acid composition because of the degradation of polyunsaturated acids (linoleic acid and linolenic acid). In addition, important losses in tocopherols content (tocopherols disappeared almost completely after storage), which has been reflected in the deterioration of the quality and antioxidant properties of oils as a function of time storage and temperature (60 °C). This fact suggests that α-tocopherol plays an important role as an antioxidant in the induction period of oxidation. The kinetic-thermodynamic study confirmed the oxidation impact on oil's quality. Indeed, the Rancimat results showed that this method could be used satisfactorily to further identify and discriminate EVOOs (the thermodynamic kinetic approach, requires only the assessment of oxidative stability), allowing to consider the latter's possible use as a preliminary but practical tool for predicting the shelf life of olive oils.

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Conflicts of Interest

The authors declare no conflict of interest.

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