



HAL
open science

Walnut (*Juglans regia* L.) oil chemical composition depending on variety, locality, extraction process and storage conditions: A comprehensive review

Catherine Rébufa, Jacques Artaud, Yveline Le Dréau

► To cite this version:

Catherine Rébufa, Jacques Artaud, Yveline Le Dréau. Walnut (*Juglans regia* L.) oil chemical composition depending on variety, locality, extraction process and storage conditions: A comprehensive review. *Journal of Food Composition and Analysis*, 2022, 110, pp.104534. 10.1016/j.jfca.2022.104534 . hal-03631908

HAL Id: hal-03631908

<https://hal.science/hal-03631908>

Submitted on 6 Apr 2022

HAL is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.

Walnut (*Juglans regia* L.) oil chemical composition depending on variety, locality, extraction process and storage conditions: A comprehensive review

Catherine Rébufa^{*}, Jacques Artaud, Yveline Le Dréau

Aix Marseille Univ, Avignon Univ, CNRS, IRD, UMR IMBE 7263, Marseille, France

ARTICLE INFO

Keywords:

Juglans regia L.
Oil chemical composition
Geographical origin
Cultivars
Extraction processes
Stability and quality parameters

ABSTRACT

This review presents a comprehensive synthesis of walnut oil chemical composition data (major and minor compounds) collected from the literature according to the varieties and the geographic origins of walnuts, and to the oil extraction processes. Considered as influencing factors, pre- and post-harvest treatment parameters of in shell-walnut and kernels were identified and works on oil stability were discussed according to walnut pre-treatment, oil extraction processes and their storage conditions. Most studies dealt with the quantification of fatty acids and tocopherols. There has been less interest in the triacylglycerol composition and minor components (sterols, volatiles, phenolic compounds) of the oil. The volatile compound profiles were mainly determined to define the oxidative degree of oil and to better understand the effect of the fatty acid degradation on the organoleptic properties of oil. Unfortunately, sensory analyses were rarely performed. This review highlights a lack of knowledge on walnut sampling before oil analysis and a distortion between some results of chemical compounds. Impact of walnut variety, walnut storage and oil extraction process was documented but few studies have considered the temporal factor of harvest, the packaging and the storage conditions of oil, which influence its chemical composition and consequently its nutritional and taste qualities.

1. Introduction

Walnut is probably one of the oldest trees providing humans with food. Known in Persia since 7000 BCE, it is native to Central Asia and it has spread through human migrations from many areas of refuge after the Last Glacial Maximum (Bernard et al., 2018).

Walnut tree belongs to the family *Juglandaceae*, genus *Juglans* and species *regia*. *Juglans regia* L. is the only species belonging to the *Dioscaryon* section (Bernard et al., 2018; Caspermeyer, 2019). It is a diploid, monoecious and dichogamous tree, wind pollinated and highly heterozygous (Feng et al., 2018). Walnut (*Juglans regia* L.) is commonly named Persian walnut, white walnut, English walnut, or common walnut (Jaiswal and Tailang, 2017). Nowadays, walnut tree is widely disseminated and cultivated in many temperate regions of Europe (since 1000 BCE), North and South America, South Africa, Asia, Australia and New-Zealand (Bernard et al., 2018). Common walnut is an economically important hardwood tree species cultivated worldwide for its high-quality wood and edible kernels (Feng et al., 2018).

Juglans regia has been the subject of several reviews of: (i) the traditional uses, phytochemistry and pharmacology of walnuts (Jaiswal

and Tailang, 2017), (ii) the antioxidant potential of fruit and tree (Jahanban-Esfahlan et al., 2019; Nguyen and Vu, 2021), (iii) the chemical composition and nutritional properties (Maestri et al., 2020; Yang et al., 2018), (iv) the applications of infrared and Raman spectroscopy for the characterisation of many nut species, the rapid estimation of the fatty acid profile, moisture content, nut quality (internal damage, fungal and insect infection) and the discrimination of cultivars (Teixeira and Sousa, 2019), (v) the walnut genetic resources and composition as well as by-product obtainment and characteristics, including oil (Martínez et al., 2010), (vi) the roasting effect on the quality and safety of oilseed oils (Zhang Y. et al., 2021).

Walnut oil, long regarded as a by-product of the processing of walnuts into kernels, has recently become a high-value product, sought after by discerning consumers. Walnut oil, strongly linked to a famous gastronomic heritage and a particular know-how from the walnut-production regions, sometimes benefits from a protected designation of origin. In addition, the growing consumer expectation for healthy lipids contributes to the edible oil market's orientation in an increasingly scientific nutritional context. Périgord walnut oil is the first walnut oil to benefit from a Controlled Designation of Origin (AOC) (JORF n°0271, 2018) and a Protected Designation of Origin (Official Journal of

^{*} Correspondence to: Aix Marseille Univ, Univ Avignon, CNRS, IRD, IMBE UMR 7263, Pôle de l'Etoile. Equipe Biotechnologie et Chimométrie, Case 451, 13397 CEDEX 20 Marseille, France.

E-mail addresses: c.rebufa@univ-amu.fr (C. Rébufa), jacques.artaud@univ-amu.fr (J. Artaud), yveline.le-dreau@univ-amu.fr (Y. Le Dréau).

Nomenclature			
AOC	Controlled Designation of Origin	KOH	Potassium hydroxide
EtOH	Ethanol	MS	Mass Spectroscopy
FA	Fatty Acid	MUFA	MonoUnsaturated Fatty Acid
FAME	Fatty Acid Methyl Esters	NaOH	Sodium hydroxide
FID	Flame Ionization Detector	OD	Ortho-Diphenolic compounds
GC	Gas Chromatography	PDO	Protected Designation of Origin
HPLC	High Pressure Liquid Chromatography	PUFA	PolyUnsaturated Fatty Acid
HS-SPME	Headspace-Solid-Phase Micro-Extraction	SBE	Subcritical Butane Extraction
ICP-AES	Inductively Coupled Plasma - Atomic Emission Spectroscopy	SC-CO ₂	Supercritical Carbon Dioxide Extraction
ICP-MS	Inductively Coupled Plasma – Mass Spectrometry	SFA	Saturated Fatty Acid
		T	Tocopherol
		T3	Tocotrienol
		TP	Total Phenolic compounds

The European Union, 2020). In 2020, the Swiss Federal Office of Agriculture (OFAG, 2020) certified that the Vaud Walnut Oil now benefits from a Protected Designation of Origin (PDO).

In the objective to consider walnut oil as healthness edible oil, highlight the factors really impacting oxidative stability is crucial for a better quality-control of walnut oil. Indeed, focus on some walnut varieties with high yield to obtain oils with a high antioxidant and healthy fatty acid potential, ensure the walnut conservation before the oil extraction process implementation, control the oil storage conditions and choose an appropriate oil extraction process to maximize oxidative stability of oils is the good approach. However, with the biodiversity and the local economy preservation in mind, some walnut varieties, not exactly fitting with these targets, must not be discarded.

In the present review, a bibliometric approach made it possible to build a database from which it was done a wide-ranging synthesis work on literature results concerning the quantification of major and minor chemical compounds of studied walnut oils. The aim of this review is to summarize the available information about the differences in walnut oil composition according to influencing factors, such as walnut sampling knowledge (variety, geographic origin, ripeness stage and growing conditions), walnut kernel pre-treatment and storage, oil extraction process and analytical techniques of quantification. This will give a better understanding of the variability of chemical composition of walnut oils and to adopt (i) the appropriate way for the walnut growing, their harvesting and their conservation, (ii) to choose the best oil

extraction process to assure a good temporal oil stability, so a healthy quality oil.

2. Bibliometric study

Bibliometric research in Web of Science Core Collection, between January 1975 and October 2021, based on the keyword “walnut” in the topic led to 8939 papers and to 4652 papers when the research was limited to title field. When the keyword “*Juglans regia*” was added, the number of papers decreased greatly to 1992 and 732, and then to 263 and 27 respectively with the keyword “oil” over-added (Fig. 1). Fifty one percent of the 263 papers were published in the category “Food Science and Technology”, 18% in “Applied Chemistry” and “Horticulture” and 10% in “Nutrition Dietetics”. About twenty papers (approximately 8%) were recorded in “Agriculture multidisciplinary”, “Plant Sciences” or “Agronomy”. Most of the papers (n = 240) were scientific articles; thirteen were referenced as proceedings papers, nine of them were reviews and only one book was counted. The most publishing countries were China (n = 60), Turkey (n = 44), Iran (n = 23), USA (n = 21) and Spain (n = 21). This great scientific interest for walnuts may be explained by the fact that China, USA and Iran are not only the main walnut-consuming countries but are also, alongside Chile and Ukraine, among the top five of the walnut-producing countries, that contribute for almost 90% of world production. France, India, Moldova, Romania and Turkey completed the top ten. Four European countries (France,

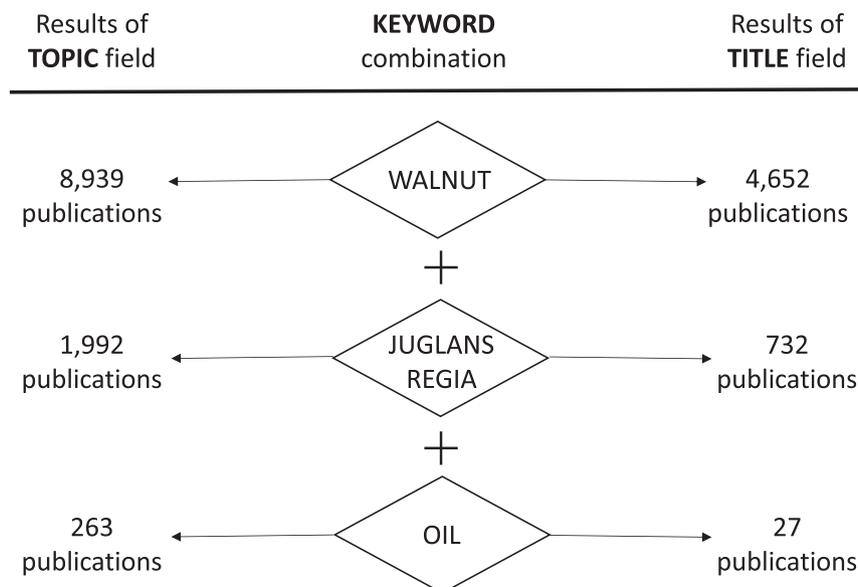


Fig. 1. Publication scores from bibliometric research in Web of Science Core Collection, between 1975 and October 2021.

Germany, Italy and Spain) and three Asian countries (India, Japan and South Korea) are also major consumers of walnuts, but their scientific production was lower than ten articles during the time period studied, except for Spain. In fact, as Spanish authors published a lot of papers on olive oil, walnut oils were also studied for the sake of comparison. French, German and Italian scientific productions were low: these countries were cited 3, 5 and 7 times, respectively, in this bibliometric study. Less than fifteen papers were published annually until 2016, then due to a growing interest for walnut oil, number of papers has noteworthy increased to reach 35 scientific papers published in 2020. Among the papers extracted from this bibliometric study ($n = 263$), 154 were selected to build this review on walnut oils, in which a particular attention was given to their composition in major and minor compounds. The distribution of these publications according to the compounds of interest is given in Fig. 2. Many publications dealt with the quantification of fatty acids because of their importance for the nutritional properties of walnut oils. Tocopherols, phenolic compounds and volatile compounds were often studied because of their antioxidant potential (tocopherols and phenolic compounds) and their important role in the sensory properties of oil (phenolic compounds and volatile compounds). The other compounds mentioned were the subject of less than five publications because of the lower interest for walnut oil in the past compared to other widely consumed vegetable oils.

3. Walnut sampling

Generally, the variety was well described. However, as many articles were related to the study of weakly commercialised but promising varieties or to the strategy of choosing genotypes obtained by hybridisation, the sample names were codified, without reference to known varieties, described and listed. Often, single or few samples per variety were considered. Three types of walnut samples were used: (i) commercially purchased samples (Esteki et al., 2017; Gao et al., 2018; Kesen et al., 2018; Matthäus et al., 2018; Ojeda-Amador et al., 2018; Persic et al., 2018; Poggetti et al., 2018); (ii) samples collected from private plantations with commercial vocation; (iii) samples from experimental orchards (walnut germplasm collections, (Greve et al.,

1992)) walnut germplasm nursery, (Li et al., 2017); walnut orchard genebank (Ozcan and Arslan, 2011); University experimental implementation area, (Savage et al., 1999; Bujdosó et al., 2016; Beyhan et al., 2017; Kafkas et al., 2017, 2020); agronomical institute (Abdallah et al., 2015; Christopoulos and Tsantili, 2015; Rabadán et al., 2018a, b, c, d; Cittadini et al., 2020); a natural walnut-fruit forest (Erturk et al., 2020). Very few studies considered sampling over several harvest years, to account for the annual variability in chemical characteristics (Amaral et al., 2004; Zhai et al., 2015; Leahu et al., 2016; Peng et al., 2017; Matthäus et al., 2018; Poggetti et al., 2018).

The quantities of nuts harvested per sample varied widely. Some authors made sure they had enough to carry out numerous analyses while others were concerned about the representativeness of their sampling, with walnut fruits randomly harvested from several trees and with at least three replicates. In some cases, all collected samples were fully mixed. The amount of walnuts harvested ranged from a few hundred grams (Dogan and Akgul, 2005; Leahu et al., 2013; Christopoulos and Tsantili, 2015; Liu et al., 2020), a few kilograms (Amaral et al., 2005; Pereira et al., 2008; Verardo et al., 2009; Bada et al., 2010; Rabrenovic et al., 2011; Zhai et al., 2015; Kodad et al., 2016; Sena-Moreno et al., 2016; Esteki et al., 2017; Rabadán et al., 2018b; Viera-Alcaide et al., 2019) up to 10 kg or more (Torres et al., 2005; Matthäus et al., 2018). Walnuts were harvested at optimum maturity stage, hand-picked from the ground or the tree. When they could not extricate themselves naturally from their green husks, they were retrieved either manually or sometimes mechanically, (Liu et al., 2020). They were often rinsed or washed either with tap water or distilled water.

The walnuts were either sun-cured or dried at room temperature, or in an oven (at 30 °C, 40 °C or 45 °C), or in a bin, or a forced-air drier (at 30 °C or 37 °C) for a defined time of 24 h to 3 days or until the humidity reached a threshold value (8–10%) or the weight a constant value. Typically, the walnuts in their shell were then stored in the dark, in paper bags or in fillets, or in plastic bags sealed or not, either at room temperature or at controlled temperatures (4 °C or 12 °C) or controlled humidity (60%) before use (Lavedrine et al., 1997; Zwartz et al., 1999; Amaral et al., 2003, 2004; Bada et al., 2010; Rabrenovic et al., 2011;

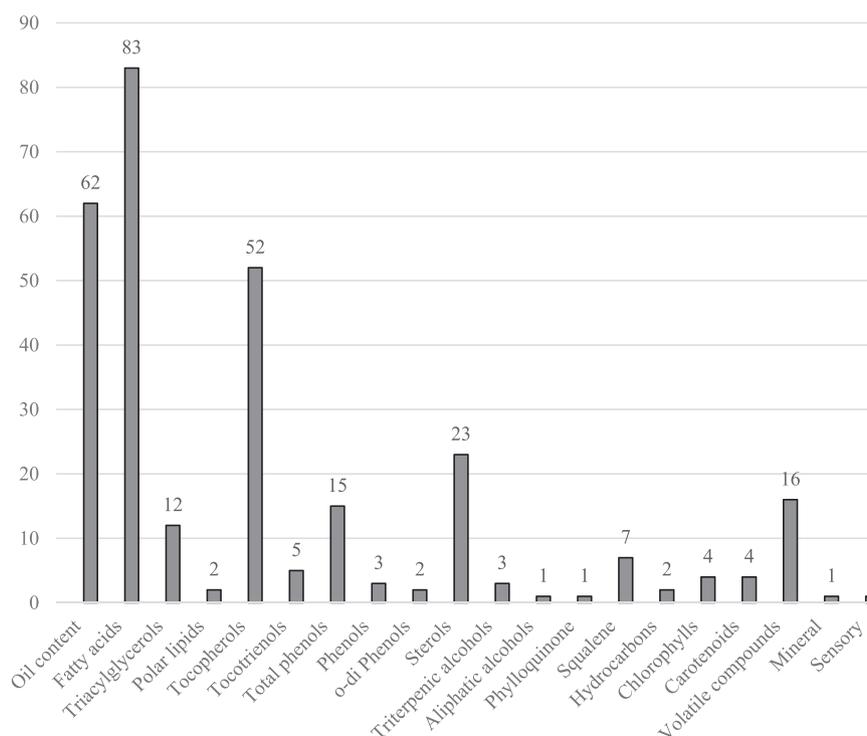


Fig. 2. Number of papers by oil content, compounds and sensory approach.

Gharibzahedi *et al.*, 2014; Zhai *et al.*, 2015; Bujdosó *et al.*, 2016; Kodad *et al.*, 2016; Poggetti *et al.*, 2018; Rabadán *et al.*, 2018b). Then, walnuts were manually cracked and shelled, and the kernels were stored in sealed plastic bags (Greve *et al.*, 1992; Özcan *et al.*, 2010; Sena-Moreno *et al.*, 2016; Li *et al.*, 2017; Poggetti *et al.*, 2018; Rabadán *et al.*, 2018c; Gao *et al.*, 2019b; Cittadini *et al.*, 2020; Liu *et al.*, 2020), vacuum packed (Savage, 2001) or in impermeable pouches flushed with dry air, N₂ or CO₂ (Christopoulos and Tsantili, 2015), or in clear or amber bottles (Özcan *et al.*, 2010; Matthäus *et al.*, 2018), in the dark either at room temperature, or refrigerated between – 4 °C and – 20 °C before analysis. Sometimes, walnuts being studied were freeze-dried (Pycia *et al.*, 2019a).

According to these previous references, different sample preparations were identified. Before analysis or before extraction of the oil, the walnut kernels were smashed, finely chopped and ground, using either a stainless-steel knife, a mortar, a coffee mill or an electric grinder. In some cases, the powder was sifted through manual or mechanical sieve. Sometimes, the powder samples were then stored in amber bottles or sealed plastic bags at 4 °C or frozen at – 20 °C. In some studies, dealing only with oil analysis, it was specified that the oils were stored in dark bottles at 4 °C or – 18 °C.

A good knowledge of the walnut sample characteristics (representativeness, varietal origin, geographical origin, agro-climatic conditions, maturity, number of harvest years, time and conditions of storage before analysis) is the key to the validity and interpretation of the analyses. In studies focusing on the chemical characterisation of walnuts and their oils, some authors endeavoured to give as much information as possible about the samples they studied, while in other articles little information was available. The walnut oil chemical data of the literature to be favoured are those for which the sampling is precisely described. On the other hand, when sampling information is not provided, chemical data should be not considered.

4. Harvest and storage conditions of walnuts and walnut kernels

To determine the best time to harvest walnuts, the effects of different ripening stages were studied to investigate the best kernel quality characteristics (lipid content, mineral composition, sensory properties, antioxidant and antibacterial activity). Some authors showed that the highest content of phenols and biologically active compounds imparting high health benefits was observed in the first stage of maturation (Amin *et al.*, 2017).

Walnut kernels are rich in polyunsaturated fatty acids vulnerable to oxidation, despite a natural antioxidant content which decreases during storage. A drying treatment was often used before storage to ensure a low residual moisture to prevent bacterial growth. Furthermore, some authors evaluated the cold storage tolerance of various cultivars according to the terminal browning index, acid value and sensory quality (Yuan *et al.*, 2019). When walnuts were stored whole or shelled at low temperatures (1 or 8 °C) directly after harvest, no negative effects were recorded, except a slight surface browning (Christopoulos and Tsantili, 2012). Although the skin of the walnuts darkens following pre- and post-harvest treatments, the dark kernels, undesirable by consumer, remain edible with oxidation parameters acceptable at the end of 6-month storage even in the packages with air and at non-refrigerated conditions (Yildiz and Karaca, 2021).

Various thermal treatments (dry heating, microwave heating and steam heating) were investigated through the lipoxygenase activity to delay the onset of oxidative rancidity and thus extend walnut shelf-life: enzyme deactivation was obtained after 30 min of dry heating (at 120 °C) or after 12 min of microwave treatment (Wang *et al.*, 2014). The effects of heat and freeze drying on the nutritional qualities of walnuts were compared; heat drying at 105 °C was recommended as an optimal drying process (Han *et al.*, 2019). It was reported that the colour deterioration and rancidity of the kernel, linked to oxidation processes, could be reduced by low-temperature handling and low-oxygen

packaging. Storage experiments under reduced oxygen atmosphere (achieved using oxygen absorbers) showed a beneficial effect (Ortiz *et al.*, 2019). Antioxidant losses and browning were prevented for kernels stored at low temperature in packaging under N₂ or CO₂, comparatively with kernels stored at 20 °C under air, and this for all the studied varieties (Christopoulos and Tsantili, 2011). Temperature of storage has the strongest consequence on walnut quality, followed by the oxygen barrier effect provided by the packaging, then lighting exposure. Stored at 20 °C in the dark in pouches having an effective oxygen barrier effect, walnuts were able to retain acceptable quality for at least 12 months (Mexis *et al.*, 2009). Alternatively, vacuum combined with iron deoxidizer package (blocking oxygen at the source) made the most significant contribution to the long storage of walnuts (Hao *et al.*, 2020).

Other authors proposed to store walnuts using edible coatings (methyl cellulose showed a remarkable superiority), considered as an alternative way for the food industry to extend the sensory stability of walnuts. Indeed, edible coatings can reduce oxygen and moisture migrations and improve the physico-chemical properties of the food (Grosso *et al.*, 2017, 2018). When walnuts were kept in the form of a paste, the optimal paste was obtained by milling roasted kernels at 110 °C for 10 min and by adding biopolymers to limit oxidation and stabilise the fat matrix, before the freeze-drying process (Dordoni *et al.*, 2015).

5. Modes of walnut oil extraction

The walnut oil extraction processes used differed according to the research objective: (i) determination of walnut oil content, (ii) selection of certain compounds of interest, (iii) implementation of new systems in industrial installations to increase oil yield, to obtain oil free from oxidized products or to promote an eco-friendly technique. Often, the effects of different extraction processes on the nutritional properties and antioxidant activities of walnut were compared. Typically, three processes were used: solvent extraction, pressing, or super critical extraction.

For the sake of simplicity, or for its better selectivity toward lipids, solvent extraction was often used to obtain walnut oil and analyse it chemically. Results differed depending on the combination of organic solvents used for the extraction (Crowe *et al.*, 2002; Miraliakbari and Shahidi, 2008a). The choice of solvents affected the minor component composition more than the fatty acid composition. It was concluded that extraction using a water / methanol / chloroform ternary mixture provides oils with higher amounts of antioxidant components than cold maceration in hexane (Gharibzahedi *et al.*, 2013). To develop an effective and eco-friendly technique for walnut oil extraction, a surfactant and salt-aided aqueous extraction method was also used to break the emulsion (Geng *et al.*, 2020), with an oil recovery rate greater than 90%, and a possibility of reusing the demulsifier. An enzyme-assisted aqueous procedure was optimised to extract oil from kernels and obtain walnut oils of higher quality, in terms of oxidative stability and tocopherol content, than organic solvent extracted oils (González-Gómez *et al.*, 2019). At the present time, the optimisation of solvent extraction processes to increase yield performance is no longer studied, because consumers are increasingly looking for less processed products. Besides, oils obtained by solvent extraction cannot benefit from the designation "virgin oil". Then, it is sometimes stressed that this technique should be replaced. Pressing extraction is the method that seems to best meet consumers' expectations. Thus, several studies were conducted to highlight the parameters to obtain the best oil with the best yield and prevent the loss of compounds with nutritional value. Different parameters of the screw pressing process were optimised at the industrial scale (Dusek *et al.*, 2017; Martínez *et al.*, 2017). Seeds with a moisture content about 7.5% pressed at a pressing temperature of 50 °C gave an oil whose quality remained comparable to that of cold-pressed seeds (Martínez *et al.*, 2008). Even though screw pressing is considered a cold process, the nozzle where oil separation occurs must be pre-heated to

obtain a satisfactory oil production. [Rabadán et al. \(2018c\)](#) studied the effect of the heating temperature and the rotational speed on the oil composition. They concluded that the rotational speed had a larger effect on the oil temperature than the nozzle temperature and that it modified the physicochemical quality parameters without affecting the fatty acid and sterol contents. Hydraulic press and screw press were also compared ([Sena-Moreno et al., 2016](#)). Because of the heating of the nozzle, a slightly greater oil yield was obtained with the screw press. The chemical composition and the values of the regulated quality parameters of the oils obtained by these two processes exhibited similar values. However, authors recommended the hydraulic press as the more appropriate method to preserve oil stability and sensory attributes. Because the Soxhlet apparatus extracted oils had lower linoleic acid and tocopherol contents than the cold pressing extracted oils, Soxhlet apparatus was less suitable for oil extraction, especially since it may lead to a chemical contamination ([Ahmed et al., 2019](#)).

The supercritical carbon dioxide (SC-CO₂) fluid extraction method seems to have a promising future. It is the most rapid and efficient method, which has the advantage of being solvent-free and requiring very little sample preparation (grinding of the nuts and a little clean-up). Nevertheless, it could have the disadvantage of (i) losing the volatile compounds during oil extraction and requiring specific extraction conditions (temperature, pressure and time) for each sample ([Crowe et al., 2002](#)), (ii) significantly decreasing the stability of the extracted oil with respect to that of press-extracted oils, in a possible relation with the oxygen content of the CO₂ used for the extraction ([Martínez et al., 2008](#)), (iii) being dependent on the particle size, which, along with operating pressure and operating temperature, has a critical effect on the extraction yield. An increase in particle size was accompanied by a decrease in solubility and yield of oil because of the increase in the resistance to mass transfer between the surface of the walnut kernel and CO₂ gas ([Salgın and Salgın, 2006](#)). The SC-CO₂ extracted oil presented a larger tocopherol content than cold hexane extracted oils, and also was clearer, showing some refining ([Oliveira et al., 2002](#)). The SC-CO₂ extraction time was significantly reduced when a CO₂/ethanol mixture was used because of the higher solubility of oil in this solvent - which had the favourable advantage of being a green solvent ([Salgın and Salgın, 2006](#)). Compared to common extraction processes (hexane extraction, cold pressing with and without oven-roasting of walnut kernels and SC-CO₂ extraction), subcritical butane extraction (SBE) was proposed as a safe, efficient, and environmentally friendly process in which the solvent was completely removed by system depressurisation. SBE could be a green production route for high-quality and high-nutritional-value walnut oil, yield and nutritional component content being however lower than with hexane extraction ([Gao et al., 2021](#)).

6. Walnut oil stability

Walnut oil is easily oxidized because of the important percentage of polyunsaturated fatty acids (linoleic and linolenic acids) ([Savage et al., 1999](#)). The studies were performed according to the oxidative stability or the antioxidant capacity.

Their oxidative stability differed from one variety to another depending on their phenol and tocopherol contents ([Amaral et al., 2003](#); [Ojeda-Amador et al., 2018](#); [Grilo and Wang, 2021](#)). However, the content of phenolic compounds in the oil is low compared to that of the kernels ([Slatnar et al., 2015](#)). The oxidative stage of oil was often evaluated by measurement of peroxide index value ([Gao et al., 2019c](#)). Based on peroxide value measure, [Nosenko et al. \(2019\)](#) determined an induction period (defined as the start of an increase of the peroxide index) and a self-life (period of peroxide value reaching 10 mequiv O/kg oil), estimated of 56 and 90 days respectively, despite a high content of polyunsaturated fatty acids. To evaluate rapidly the oxidation stage of oil sample, a Partial Least Square model allowed predicting the peroxide value of virgin walnut oils from Fourier Transform Infrared profiles ([Liang et al., 2013](#)). One study focused on the influence of various

storage conditions on peroxide value and the walnut oil colour. Six months of storage induced an increase of peroxide value but changes in the oil colour have not been significant enough ([Leahu et al., 2016](#)). The Rancimat method, an accelerated ageing test, measures the oxidation resistance of oil by defining an induction period (time from which secondary oxidation products appear) ([Gao et al., 2021](#); [Grilo and Wang, 2021](#); [Matthäus, 1996](#); [Slatnar et al., 2015](#); [Zhang L. et al., 2021](#)). This method showed that walnut oils with a higher oleic acid content had the longest shelf life ([Zhang L. et al., 2021](#)). The reduced stability of oils, measured by the Rancimat method (3.9 to 7.8 h), appeared to be correlated to higher levels of linolenic acid ([Savage et al., 1999](#)). An induction period identical of that measured by Rancimat method has been obtained from chemiluminescence measurements ([Matthäus, 1996](#)).

The antioxidant capacity of oils was evaluated with different methods: diphenyl picrylhydrazyl radical scavenging method (DPPH), ferric reducing antioxidant power (FRAP) assay, azino-bis-(ethylbenzothiazoline-sulfonic acid) (ABTS) assay, and oxygen radical absorbance capacity (ORAC) assay. A multi linear regression model allowed to highlight the contribution of C16:0, C18:0 and C18:3w3 fatty acids, SOS, PLL and LLL triacylglycerols, D5-avenastérol and g-tocopherol to the antioxidant capacity of walnut oil extracted with various extraction processes ([Gao et al., 2021](#)).

Roasting kernels, used to increase oil yield, could contribute to influence the oil stability. [Zhang Y. et al. \(2021\)](#) reviewed the influence of three roasting process (oven, microwave and coffee bean roaster) on oil quality and chemical composition. It was noted that the fatty acid content was weakly impacted. Upon oven roasting walnut kernels, the peroxide value, and the content of tocopherols, phytosterols, and polyphenols increased whereas squalene content decreases. Microwave roasting induced a significant decrease the content of tocopherols and sterols but improved the flavour and oxidative stability. Coffee bean roaster induced an increase of peroxide value and the conjugated dienic acid content and decreased tocopherol content.

The oxidative stability and the oxidative capacity of oil were also discussed according to the extraction mode: maceration in solvent, cold solvent extraction, Soxhlet method, cold press process, and SC-CO₂ extraction ([Crowe and White, 2003](#); [Gharibzadeh et al., 2014](#); [Copolovici et al., 2017](#); [Ahmed et al., 2019](#)). The polarity of solvent used for oil extraction mainly influences the extraction of the antioxidant species. Indeed, walnut oil obtained by acetone extraction had a relatively weak phytosterol content but higher polyphenol content, which contributed to its better antioxidant capacity compared with the oils obtained by hexane, ethyl acetate, petroleum ether or chloroform/methanol (v/v, 2/1) ([Gao et al., 2019b](#)). Some results showed that chloroform/methanol-extracted oils were more stable than hexane-extracted oils in both the accelerated autoxidation and photo-oxidation studies ([Miraliakbari and Shahidi, 2008b](#)). For the same walnut variety, the hexane extracted oil showed the best oxidative stability index and free radical scavenging capacity, compared to cold pressing, roasting pressing, SBE or SC-CO₂ extracted oil, perhaps because of its highest phenolic content ([Gao et al., 2021](#)). The antioxidant activities and the total phenol contents were found to be lower in cold-pressed oils than solvent extracted oils using a Soxhlet apparatus. However, Soxhlet extracted oils contained higher amounts of total tocopherols (known for their antioxidant activity) than cold-pressed oils ([Rabrenovic et al., 2011](#); [Ahmed et al., 2019](#)). Oxidative stability indices and tocopherol contents were significantly lower in SC-CO₂ extracted oil than in pressed oil. Moreover, when stored in darkness, SC-CO₂ extracted oil was less stable than pressed oil, but exhibited greater photo-oxidative stability, possibly because of the absence of chlorophyll in SC-CO₂ extracted oil while detected in the pressed oil ([Crowe and White, 2003](#)).

Various studies reported the effectiveness of the addition of antioxidant compounds to extend the shelf life of walnut oil. Some authors emphasised the potential value of acidic amino acid residues for the

protection of the unsaturated fatty acids from oxidation (Jie et al., 2019). Martínez et al. (2013) evaluated the combined effects of storage conditions (six months at room temperature, under fluorescent light or in darkness) and addition of natural and synthetic antioxidants (rosemary extract, ascorbyl palmitate and Tert-butylhydroquinone). They showed that walnut oil was highly susceptible to free-radical oxidation (autoxidation) even under relatively mild temperature conditions (25 ± 1 °C). Without addition of any antioxidant, the oils stored in darkness and the oils stored in the light oxidised at a similar rate. By contrast, the addition of antioxidants (rosemary extract alone or combined with the synthetic antioxidants) significantly reduced lipid oxidation under darkness conditions but increased the formation of primary and secondary oxidation products in the light exposed oils (Martínez et al., 2013). As reported by Bakkalbaşı (2019), phenolic extracts obtained from ultrasound applied oil with added press-cake (200 mg/kg oil) can be successfully used as natural antioxidants to improve the oxidative stability of walnut oil. An addition of 0.005% of lycopene improved walnut oil quality and extended walnut shelf life up to 16 months (Xie et al., 2018). The immersion of herbs (sage and oregano; 3%, 5%, and 7% (w/w) herb/oil; 30 °C for 10 days) increased the oxidative stability of the investigated oils, and particularly oils rich in polyunsaturated fatty acids (Somogyi et al., 2015). When sage, thyme and basil (under different forms: aerial plant intact or part cut into pieces, or leaves only) were added to walnut oil, an increase of the antioxidant activity of walnut oil was mainly observed with sage addition (Laskos et al., 2021).

7. Oil content of walnut kernels

To quantify walnut oil content, numerous authors used official standard methods, such as the Association of Official Analytical Chemists methods (Helrich, 1990; Cunniff, 1995; Horwitz and Latimer, 2005), International Organization for Standardization methods (ISO 659, 1998; ISO 734-1, 1998), or the Official Methods and Recommended Practices of the American Oil Chemists' Society (AOCS Official Method Am 2-93, 1997) and derived methods. They included traditional Soxhlet extraction with conventional solvents, such as petroleum ether, diethyl ether or hexane generally boiling for 6 h, but some procedures lasted from 1 h (Beyhan et al., 2017) to 24 h (Amin et al., 2017). Verardo et al. (2009) used a Soxtec™ extraction unit, a fully automated system for fast and safe extraction with diethyl ether solvent. Some authors performed cold lipid extraction with successive extractions using hexane (Miraliakbari and Shahidi, 2008a) or hexane/isopropanol (3:2, v/v) at room temperature under vigorous stirring for 1 h (Maguire et al., 2004) or chloroform/methanol (1:1, v/v) with homogenisation at 8000 rpm for 3 min (Miraliakbari and Shahidi, 2008a). A nuclear magnetic resonance (NMR) method was developed by Poggetti et al. (2018) to quantify the oil content of 228 walnut samples after validation by Soxhlet extraction.

Comparison of the results was difficult because the unit used to express fat content differed among studies: % without any precision, % or g/100 g of kernel without precision (fresh or dried), % of fresh matter or fresh weight and g/100 g dried kernel or dried matter. Thus, only results of oil content expressed from dried kernels or fresh kernels (moisture corrected) were considered for comparison. They related to cultivars or genotypes from thirteen countries: Argentina (Martínez et al., 2006; Martínez and Maestri, 2008; Cittadini et al., 2020), China (Liu et al., 2020; Bingqing et al., 2021), Greece (Christopoulos and Tsantili, 2015), Hungary (Bujdosó et al., 2010, 2016), Italy (Verardo et al., 2009), Iran (Gharibzadeh et al., 2014), Kyrgyzstan (Erturk et al., 2020), Marocco (Kodad et al., 2016), New Zealand (Zwarts et al., 1999; Savage, 2001), Poland (Pycia et al., 2019a, 2019b), Portugal (Amaral et al., 2003; Pereira et al., 2008), Romania (Leahu et al., 2013), Spain (Sánchez-Morgado et al., 2013; Tapia et al., 2013; Viera-Alcaide et al., 2019), Turkey (Koyuncu et al., 2001; Dogan and Akgul, 2005; Ozkan and Koyuncu, 2005; Özcan et al., 2010; Ozcan et al., 2020) and USA (Kafkas et al., 2020; Grilo and Wang, 2021). Considering only the results for walnuts collected at maturity, the average value of oil content was

66.59% of dried kernel, the minimum oil content was 43.3% of dried kernel (Howard variety, (Grilo and Wang, 2021)) and the maximum oil content was 78.48% of dried kernel (Tulare variety, (Cittadini et al., 2020)). Cold hexane extraction gives the highest oil yield over cold pressing, SBE, roasting pressing and SC-CO₂ respectively (Gao et al., 2021). The nature of the solvent used for the cold solvent extraction method also influenced the oil yield. According to Miraliakbari and Shahidi (2008b) and Gao et al. (2019b), solvents like ethyl acetate, petroleum ether and chloroform/methanol are more efficient than hexane or acetone for extracting walnut oils. Nevertheless, not all these solvents are authorized in food industry by most of the legislation worldwide.

Different researchers studied the influence of the maturing stage, harvest date, growing conditions and latitude on the oil content. The evolution of the oil content during the walnut ripening was studied at three stages: at packing-tissue-brown (PTB, i.e., stage where packing tissue between and around the kernel halves just turned brown), pre-PTB and post-PTB (i.e., walnuts harvested 10 days earlier than and later than PTB point, respectively). The oil content increased with the ripening, from 51.8 to 58.3 g/100 g of kernel (Amin et al., 2017). Pycia et al. (2019b) collected three walnut varieties at three different maturity stages. An increase ranging from 14 to 18 g/100 g between the first and the last stage of maturity was recorded. However, oil contents they recorded (24.25 g/100 g dried matter) were meaningfully lower than those usually reported in the literature (Pycia et al., 2019a). Koyuncu et al. (2001) confirmed that the oil content of a same walnut variety increased significantly during the development of the kernels. Other authors examined the evolution of the oil content according to the date of harvesting (Matthäus et al., 2018). The oil content increased from 39.1 to 61.8 g/100 g of kernel in walnuts harvested over one month and a half with a maximum of 70.5 g/100 g after 20-day harvest. As for the growing conditions, Verardo et al. (2009) concluded that the oil content was influenced neither by nitrogen fertilisation levels nor by irrigation water amounts. Furthermore, no significant effect of altitude was found on the oil content of walnut samples collected over two successive years from wild walnut trees, genetically characterised and growing between 1 and 1073 m altitude; no pruning, no horticultural practice (fertilisation and irrigation) and no pest inspection was carried out. The oil content range for trees grown from different accessions from one year to the next remained almost identical (56.2–71.6% w/w in 2013 and 54.2–72.2% w/w in 2014) (Poggetti et al., 2018). A study on sixteen genotypes from two natural walnut growing areas in Kyrgyzstan showed that genotypes selected from one of regions have led to a higher oil yield, but no correlation was established with the altitudes of mentioned locations (Erturk et al., 2020). A similar behaviour has been found with a Chinese variety growing on different surrounding sites (Bingqing et al., 2021). An important increase of yield was observed when kernels were roasting (120 °C during 30 min) often performed because of its desirable effects of flavours because of the water evaporation (Ghafoor et al., 2020).

8. Fatty acids

Walnut oil is claimed to be a healthy source of lipids for humans because it is rich in monounsaturated fatty acids (MUFA) - primarily oleic acid - and polyunsaturated fatty acids (PUFA) - linoleic and linolenic acids.

Generally, fatty acids were analysed by gas chromatography (GC) of the fatty-acid methyl esters (FAME) prepared mainly by direct trans esterification with KOH methanol or NaOH methanol (ISO, 5509:2000, 2000; IUPAC No. 2301, 1987) or adapted methods based either on saponification (Martínez et al., 2006; Torres et al., 2005) and esterification with BF₃ methanol (Amaral et al., 2003; Beyhan et al., 2017; Maguire et al., 2004). Methods using acids after saponification were also performed (Viera-Alcaide et al., 2019; Martínez et al., 2008). FAME analysis was carried out on GC columns from 30 to 100 m in length

coated with polar stationary phases available from different column suppliers (polyethylene glycol phase (Han et al., 2019; Wu et al., 2020) or aryl-polysiloxane phases with different percentages of cyanopropyl, cyanopropylphenyl or phenyl (Bakkalbaşı, 2019; Geng et al., 2020; Iqbal et al., 2016; Yang et al., 2018)), making it possible to achieve a more or less fine separation of the minor FAMES and of the cis/trans isomers.

Fatty acids were used by authors to characterise walnut kernels or oils for consumption. In a large part of the studies, only the content of the five major fatty acids was mentioned. Therefore, Table 1 presents the mean, minimum and maximum content of these five FA, calculated from data reported in the literature, according to the walnut variety for all geographic origins combined. They were palmitic acid (16:0), stearic acid (18:0), oleic acid (18:1 ω 9), linoleic acid (18:2 ω 6), and linolenic acid (18:3 ω 3), with mean values of 6.65% (from 2.41 to 10.41%), 2.53% (from 0.60 to 4.45%), 19.65% (from 5.70 to 31.96%), 58.91% (from 50.15 to 74.00%) and 11.44% (from 6.08 to 20.60%), respectively.

All fatty acid contents exhibited significant variations among walnut varieties. Oleic and linolenic acids were identified as markers to discriminate between walnut varieties (Martínez et al., 2006). Six additional FAs (myristic, palmitic, palmitoleic, stearic, linoleic and eicosenoic acids) were used to differentiate between oils of walnuts from Hamedan province (Iran), and only myristic acid did not exhibit any significant differences among walnut cultivars (Gharibzadeh et al., 2014).

Considering FA contents during three walnut ripening stages (at maturity, 10 days earlier and 10 days later than the maturity date), no significant difference was observed among seven SFAs except pentadecanoic acid (C15:0) which increased significantly until stage of maturity and stayed constant after (Amin et al., 2017). Similarly, total MUFA content did not statistically differ during ripening although the amount of cis-11-eicosenoic acid (C20:1c-11) significantly increased at the third maturity stage. The content in PUFAs markedly decreased as ripening advanced (Amin et al., 2017). Likewise, Matthäus et al. (2018) found high linoleic and linolenic acid contents in walnut oils from kernels in their initial stages of development, which have slightly but continuously decreased during ripening, while the oleic acid content increased and the palmitic acid content remained constant. These results confirmed those of Koyuncu et al. (2001) showing that during fruit development, the increase in the oleic acid content was synchronised with the decrease in the linolenic, linoleic and palmitic acid contents while the stearic acid content remained unchanged. So, the sum of FAs was remained almost constant as kernels developed. Nonetheless, Amin et al. (2017) suggested that the degree of unsaturation of walnut oil may be influenced by various factors, such as variety, environment, location and others.

Climatic variations and different cultivation altitudes associate with geographic origin of walnuts have been showed responsible for the fluctuations of the FA contents. A correlation between various FA contents of several genotypes of different regions of Kyrgyzstan and the agroclimatic parameters was evoked but no proved (Erturk et al., 2020). It was also found that different environmental and agronomic conditions have affected the oil FA content of a Chinese walnut variety, but no statistic treatment was performed to confirm a significant effect (Bingqing et al., 2021). A strong relationship between the geographic origin and the FA contents of walnut oils was demonstrated (Martínez et al., 2006; Akhiani et al., 2017; Esteki et al., 2017), especially for the oleic acid content strongly related with the climatic characteristics (average air temperature, namely) of the planting area (Gao et al., 2018). However, in this latest study, no characterisation of the varieties was specified, which suggests that they may not be the same from one region to another. Gu et al. (2018) found that FA content varied more according to the variety than to the geographical origin of walnuts and none of the fatty acids were identified as markers of the geographical origin of walnuts (common varieties from three areas in the Xinjiang Autonomous Region, China) (Gu et al., 2018). Other authors claimed that fatty acid stable carbon isotope ratios have been an efficient approach to differentiate the origin of walnut-producing provinces in

China (Zhang L. et al., 2021).

As it is not always possible to extract oil from walnuts immediately after harvest, some researchers focused on the effect of storage conditions on fatty acid contents. The FA contents of cold pressed oils from full maturity kernels of two varieties stored in controlled atmospheres (light, temperature and humidity) over 1 to 28 weeks were shown to be variety dependent rather than storage conditions (Grilo and Wang, 2021). When a specific kernel package was performed (air, N₂ or CO₂ and stored at 1 °C or 20 °C for 12 months), SFA (myristic, palmitic, stearic, and arachidic acids) contents remained stable, whereas MUFA and PUFA (palmitoleic, oleic, gondoic, linoleic and linolenic acids) contents decreased, except vaccenic acid content which remained unchanged. Low temperature and O₂ unavailability prevented the FA losses (Christopoulos and Tsantili, 2015). When different drying temperatures (-55 °C, 60 °C, 105 °C or 140 °C) were tested, the highest contents of stearic, arachidic, oleic, gadoleic and α -linolenic acids were detected at freeze-drying temperature. Moreover, the heating drying (at 105 °C or 140 °C) induced a negative effect on the contents of specific fatty acids and therefore on the ratio of n-6/n-3, which was found optimal for the freeze-drying treatment (Han et al., 2019). A kernel roasting (at 120 °C during 30 min), generally used to increase the oil flavours, led to an increasing of FA contents depending on the walnut varieties, because of the relative humidity removing during walnut roasting (Ghafoor et al., 2020). In oils from walnuts gamma irradiated for kernels microbial decontamination (2–10 kGy), the FA contents were affected for absorbed doses higher than 6 kGy. An increasing trend for stearic and linoleic acids and a decreasing trend of oleic acid were observed (Iqbal et al., 2016). The effect of the hulling method applied to walnuts on oil composition has been studied. The manual dehulling method induced the highest unsaturated fatty acid content compared to the natural heaping method, the method using a solid ethylene-releasing agent or the ethephon immersion method (Wei et al., 2020).

Differences in FA contents were also observed depending on the walnut oil extraction process. Despite the easy use and the ability of cold extraction to preserve walnut nutritional properties, minor differences in the FA composition were observed when walnut kernels were cold pressed under different moisture and temperature conditions (Martínez et al., 2008). Generally, FA contents, mainly the linoleic and linolenic acid contents, were found higher in cold-pressed than in Soxhlet extracted oils (Ahmed et al., 2019) as well than in cold solvent extraction (Gao et al., 2019c). However, no difference in FA composition was observed between water/methanol/chloroform extraction and cold-press extraction, but a maceration extraction procedure generated a significantly lower content of oleic acid (Gharibzadeh et al., 2013). The nature of the solvent used for the cold solvent extraction influenced the FA content. Chloroform/methanol (2/1, v/v) gave the higher linoleic acid content compared to ethyl acetate, petroleum ether, acetone and hexane. By contrast, hexane gave higher oleic acid content than the other solvents. Thus, differences in the contents of MUFA and of PUFA were observed, while the SFA contents are not modified by the nature of the solvent (Gao et al., 2019b). From one to another aqueous extraction method (aqueous extraction, surfactant or salt-aided aqueous extraction) no significant difference in the FA content was detected (Geng et al., 2020). Nevertheless, the highest values of PUFA were observed in walnut oils extracted by an enzyme-assisted aqueous methodology (41.8 °C and 88 min), while the highest amount of saturated fatty acids (SFA) was observed in hexane oil extraction by Soxhlet causing PUFA degradation (González-Gómez et al., 2019). The SC-CO₂ technology, with its undeniable advantages in terms of quality and environment protection, led to a FA composition like that obtained when the oils were extracted by Soxhlet extraction with hexane (Oliveira et al., 2002). SC-CO₂, SBE and cold pressing extracted oils had the highest PUFA contents and the lowest MUFA contents over cold hexane extracted oils and roasting pressing oils through linoleic and oleic acids, respectively (Gao et al., 2021).

Table 1
Relative percentages of main fatty acids for the studied cultivars.

Variety	Country	N	16:0				18:0				18:1 ω 9				18:2 ω 6				18:3 ω 3				Oil Ext. Mode	References
			Mean	Min	Max	Sdv	Mean	Min	Max	Sdv	Mean	Min	Max	Sdv	Mean	Min	Max	Sdv	Mean	Min	Max	Sdv		
Alsőszen-tiváni	Hungary	4	6.26	6.19	6.37	0.08	2.48	2.26	2.78	0.22	24.91	21.83	28.32	2.72	55.67	52.08	57.53	2.45	9.99	9.33	11.10	0.77	Solvent	Bujdosó et al., 2016, 2010
Ashley	USA	1	4.40				0.60				5.70				69.00				20.60				Press, Solvent	Greve et al., 1992
Baopi	China	2	5.56	5.33	5.79		2.70	1.69	3.73		17.20	14.88	19.54		65.00	62.22	67.87		9.26	7.93	10.58		Press	Zhang et al., 2021
Benhetao	China	1	4.61				2.79				18.00				66.28				7.53				Press	Zhang et al., 2021
Bilecik	Turkey	1	5.82				3.82				13.80				62.92				13.16				Soxhlet	Kafkas et al., 2017
Bofeng	China	1	6.65				2.32				22.63				57.02				10.72				Soxhlet	Li et al., 2017
Bokexiang	China	1	7.02				2.34				26.05				54.78				9.19				Soxhlet	Li et al., 2017
Bonifác	Hungary	2	6.89	6.64	7.14	0.35	2.19	1.94	2.43	0.35	20.30	19.12	21.48	1.67	57.63	56.62	58.63	1.42	11.74	11.61	11.86	0.18	Solvent	Bujdosó et al., 2016, 2010
Büyük Oba	Turkey	3	6.40	6.28	6.51	0.16	2.76	2.64	2.89	0.18	23.17	22.78	23.55	0.54	56.21	55.93	56.48	0.39	16.13	15.77	16.48	0.50	Soxhlet	Ahmed et al., 2019; Özcan et al., 2010
Chaboksar	Iran	1	11.21				2.52				23.47				51.36				10.48				Soxhlet	Gharibzahedi et al., 2014
Chandler	Argentina, China, France, Greece, Hungary, Italy, Spain, Turkey, USA	31	5.56	0.24	6.95	1.64	2.08	0.70	3.41	0.66	14.82	6.10	17.84	2.46	60.47	54.10	66.20	2.86	15.57	8.51	25.00	3.06	Press, Soxhlet, Solvent,	Greve et al., 1992; Martínez et al., 2006; Martínez and Maestri, 2008; Verardo et al., 2009; Tapia et al., 2013; Christopoulos and Tsantili, 2015; Bujdosó et al., 2016; Ojeda-Amador et al., 2018; Gao et al., 2019c; Cittadini et al., 2020; Özcan et al., 2020; Kafkas et al., 2017, 2020; Grilo and Wang, 2021
Chaotian	China	1	7.91				2.69				15.20				68.05				8.28				Press	Zhang et al., 2021
Combe	Canada	1	5.87				3.24				15.73				57.29				15.75				Solvent	Li et al., 2017
Criolla	Argentina	3	7.72	7.67	7.81	0.08	1.77	1.71	1.88	0.10	18.59	17.31	21.16	2.22	57.57	57.27	57.82	0.28	14.37	11.88	15.61	2.15	Soxhlet, Press	Torres et al., 2005; Martínez et al., 2006
Dapao	China	16	6.48	5.31	7.29	0.67	3.07	2.25	3.96	0.52	22.39	14.22	35.31	6.59	59.68	46.32	68.33	6.13	7.93	3.93	13.25	2.37	Press	Hao et al., 2020; Zhang et al., 2021
Dublin's glory	New Zealand	3	7.32	7.00	7.76	0.39	1.46	0.08	2.30	1.20	26.02	18.95	33.00	7.03	53.74	49.30	57.01	3.98	10.37	8.00	13.10	2.57	Solvent, Press	Savage et al., 1999; Zwarts et al., 1999
Durham	USA	1	6.87				2.39				20.65				60.23				9.70				Soxhlet	Kafkas et al., 2020
Esterhazy	Europe, New Zealand	3	7.82	7.30	8.70	0.76	1.61	1.50	1.70	0.10	18.38	16.50	21.20	2.49	59.04	57.60	60.70	1.56	12.68	12.10	13.54	0.76	Solvent, Press	Savage et al., 1999; Zwarts et al., 1999
Earliest	USA	1	6.95				2.40				17.47				63.11				9.88				Soxhlet	Kafkas et al., 2020
Eureka	USA	1	3.60				0.80				7.70				71.00				16.30				Press, Solvent	Greve et al., 1992
Fernette	Argentina	1	7.10				2.65				14.51				59.20				16.56				Press	Cittadini et al., 2020
Franquette	Argentina, Greece, Hungary, Italy, Portugal, Tunisia, USA	26	7.65	6.59	9.70	0.86	2.73	1.89	5.79	1.02	18.60	12.20	28.44	4.55	57.62	50.23	60.47	3.50	11.96	10.64	14.76	1.07	Solvent, Soxhlet, Press	Amaral et al., 2003; Torres et al., 2005; Martínez et al., 2006; Martínez and Maestri, 2008; Pereira et al., 2008; Bouabdallah et al., 2014; Christopoulos and Tsantili, 2015; Bujdosó et al., 2016; Sena-Moreno et al., 2016; Poggetti et al., 2018; Kafkas et al., 2020
Geisenheim	Serbia	2	7.55	7.40	7.70	0.21	1.65	1.60	1.70	0.07	19.20	18.60	19.80	0.85	57.60	57.20	58.00	0.57	13.10	13.10	13.10	-	Press	Rabrenovic et al., 2011
Hanfeng	China	1	5.36				3.00				18.37				63.47				9.47				Soxhlet	Wu et al., 2020
Hartley	Argentina, China, France, Greece, Italy, Spain, Tunisia, Turkey, USA	13	7.17	3.20	9.07	1.51	2.88	0.80	3.93	1.02	15.69	11.80	21.49	2.69	60.64	50.44	67.95	4.90	12.86	8.51	17.40	2.51	Solvent, Soxhlet, Press	Greve et al., 1992; Martínez and Maestri, 2008; Tapia et al., 2013; Bouabdallah et al., 2014; Christopoulos and Tsantili, 2015; Kafkas et al., 2017, 2020; Ojeda-Amador et al., 2018; Rabadán et al., 2018d; Poggetti et al., 2018; Gao et al., 2019c
			Mean	Min	Max	Sdv	Mean	Min	Max	Sdv	Mean	Min	Max	Sdv	Mean	Min	Max	Sdv	Mean	Min	Max	Sdv		

(continued on next page)

Table 1 (continued)

Variety	Country	N	16:0				18:0				18:1o9				18:2o6				18:3o3				Oil Ext. Mode	References
			Mean	Min	Max	Sdv	Mean	Min	Max	Sdv	Mean	Min	Max	Sdv	Mean	Min	Max	Sdv	Mean	Min	Max	Sdv		
Howard	Argentina, China, France, Italy, Spain, Turkey, USA	8	6.14	3.20	7.14	1.49	2.47	0.70	3.44	0.95	12.68	6.60	14.93	3.11	61.27	56.88	72.00	5.63	17.16	13.26	18.65	2.07	Press, Soxhlet, Solvent	Greve et al., 1992; Tapia et al., 2013; Kafkas et al., 2017, 2020; Poggetti et al., 2018; Cittadini et al., 2020; Grilo and Wang, 2021
Huget	Spain	2	6.97	6.87	7.06	0.13	3.24	3.00	3.48	0.34	22.47	20.11	24.82	3.33	57.24	55.51	58.96	2.44	9.60	8.70	10.50	1.27	Press	Rabadán et al., 2018d
Ioli cv grec	Greece	1	6.93				3.00				21.30				54.75				9.99				Press	Christopoulos and Tsantili, 2015
Ivanhoe	USA	1	7.86				2.43				16.95				59.23				13.24				Soxhlet	Kafkas et al., 2020
Jimbofeng	China	6	6.07	5.40	6.50	0.43	2.43	2.18	3.01	0.30	20.14	17.87	24.94	2.50	59.99	55.72	61.96	2.34	10.11	8.20	11.44	1.30	Soxhlet	Li et al., 2017
Jinfend	China	1	8.73				2.11				18.66				59.75				10.01				Soxhlet	Li et al., 2017
Jinlong	China	2	5.38	4.98	5.78	0.57	2.60	2.44	2.76	0.23	25.87	20.11	31.62	8.14	57.36	53.73	60.99	5.13	7.98	6.50	9.45	2.09	Soxhlet	Li et al., 2017
Jinxiang	China	1	6.28				2.63				21.77				59.55				8.78				Soxhlet	Li et al., 2017
Jizhaomian	China	1	5.92				3.10				11.59				69.47				9.92				Soxhlet	Liu et al., 2020
Jupiter	Serbia	2	6.95	6.90	7.00	0.07	1.75	1.70	1.80	0.07	23.30	22.90	23.70	0.57	58.20	58.10	58.30	0.14	9.50	9.10	9.90	0.57	Press	Rabrenovic et al., 2011
Kaman	Turkey	6	5.57	0.81	6.67	2.33	2.79	2.60	2.95	0.16	23.01	19.71	26.40	3.04	54.41	49.70	56.71	2.43	14.96	14.30	15.79	0.52	Press, Soxhlet	Özcan et al., 2010; Ahmed et al., 2019
Karaj	Iran	1	8.74				4.45				24.21				50.55				11.17				Soxhlet	Gharibzahedi et al., 2014
Lake	Canada	1	5.59				2.83				16.39				60.96				12.11				Solvent	Li et al., 2017
Laoshu	China	1	5.67				2.43				15.63				68.82				7.81				Press	Zhang et al., 2021
Lara	Argentina, Italy, Portugal	11	6.96	6.02	8.15	0.76	2.29	1.63	2.80	0.40	17.16	14.26	19.72	2.64	59.66	56.97	62.50	2.43	13.22	12.16	15.20	1.13	Solvent, Soxhlet, Press	Amaral et al., 2003; Martínez and Maestri, 2008; Pereira et al., 2008; Ojeda-Amador et al., 2018; Poggetti et al., 2018
Lauzeronne	Tunisia	1	7.36				3.26				19.94				61.83				7.61				Solvent	Bouabdallah et al., 2014
Li2	China	1	5.55				3.15				24.46				57.43				9.05				Soxhlet	Wu et al., 2020
Li1	China	1	5.52				2.88				25.77				57.81				7.65				Soxhlet	Wu et al., 2020
Liao	China	6	5.62	4.95	6.13	0.36	3.40	2.99	3.72	0.34	24.59	17.12	33.49	6.50	57.55	50.72	64.07	4.91	8.51	5.72	9.89	1.59	Soxhlet	Wu et al., 2020
Liaohe	China	4	6.09	5.91	6.45	0.25	2.79	2.32	3.18	0.36	21.64	18.90	24.22	2.36	58.74	55.55	61.21	2.37	9.93	7.27	11.93	1.96	Soxhlet	Li et al., 2017
Liaoruifeng	China	1	6.21				3.13				17.28				63.55				9.48				Soxhlet	Wu et al., 2020
Luguang	China	1	5.63				3.09				21.94				60.23				8.00				Soxhlet	Li et al., 2017
Maras-18	Turkey	2	6.78	6.57	6.98	0.29	3.65	3.59	3.70	0.08	24.30	21.02	27.57	4.63	56.52	53.42	59.62	4.38	8.19	7.83	8.55	0.51	Soxhlet	Kafkas et al., 2017
Marbot	Portugal	3	6.75	6.35	7.14	0.56	2.78	2.77	2.78	0.01	16.43	16.34	16.51	0.12	59.28	58.90	59.66	0.54	13.54	12.74	14.33	1.12	Solvent, Soxhlet	Amaral et al., 2003; Pereira et al., 2008
Mayette	Argentina, Portugal, Spain	6	6.97	5.95	7.96	0.75	2.81	1.77	3.85	0.77	21.58	16.08	28.88	4.93	54.94	50.52	57.46	2.81	12.98	8.72	17.61	3.25	Solvent, Press, Soxhlet	Amaral et al., 2003; Martínez and Maestri, 2008; Pereira et al., 2008; Rabadán et al., 2018d
Mc Kinster	New Zealand	3	7.47	6.22	8.20	1.09	1.49	0.06	2.50	1.27	18.64	18.40	18.80	0.21	60.60	60.20	61.31	0.61	10.68	10.30	11.10	0.40	Solvent, Press	Savage et al., 1999; Zwarts et al., 1999
Mellanaise	Portugal	3	6.78	6.31	7.02	0.50	2.70	2.65	2.80	0.11	15.36	14.49	17.09	1.84	60.46	58.75	61.31	1.81	13.18	12.51	14.51	1.41	Solvent, Soxhlet	Amaral et al., 2003; Pereira et al., 2008;
Meyric	New Zealand	3	7.47	7.20	7.90	0.38	1.46	0.08	2.20	1.20	20.03	18.09	23.10	2.69	57.74	55.50	59.30	1.99	12.27	11.30	13.31	1.01	Solvent, Press	Zwarts et al., 1999; Savage et al., 1999;
Midland	Turkey	1	6.41				3.86				17.63				61.22				10.33				Soxhlet	Kafkas et al., 2017
Milotai 10	Hungary	2	6.69	6.60	6.78	0.13	2.54	2.40	2.67	0.19	22.37	21.74	23.00	0.89	57.57	57.20	57.93	0.52	9.70	9.38	10.02	0.45	Solvent	Bujdosó et al., 2010, 2016
Milotai böltermó	Hungary	2	6.77	6.57	6.97	0.28	2.48	2.40	2.56	0.11	21.15	19.33	22.97	2.57	57.58	56.94	58.22	0.91	11.20	10.98	11.42	0.31	Solvent	Bujdosó et al., 2010, 2016
Milotai intenziv	Hungary	2	6.58	6.51	6.65	0.10	2.19	2.17	2.21	0.03	25.48	20.81	30.14	6.60	54.39	50.10	58.67	6.06	9.87	9.20	10.54	0.95	Solvent	Bujdosó et al., 2010, 2016
Milotai k é sei	Hungary	2	6.70	6.25	7.14	0.63	2.22	1.94	2.49	0.39	20.77	19.12	22.41	2.33	57.98	57.33	58.63	0.92	11.14	10.66	11.61	0.67	Solvent	Bujdosó et al., 2010, 2016
Nerpio	Spain	2	7.84	7.80	7.87	0.05	2.62	2.58	2.65	0.05	18.70	17.33	20.07	1.94	59.09	58.16	60.02	1.32	11.02	10.61	11.43	0.58	Press	Rabadán et al., 2018d
Niangqing	China	1	6.82				1.76				32.78				52.28				8.59				Press	Zhang et al., 2021
Parisienne	Portugal, Spain, Tunisia	6	6.83	6.16	7.37	0.55	2.92	2.41	3.44	0.37	21.49	16.49	27.46	5.16	57.15	52.49	62.45	4.48	10.90	9.49	13.20	1.87	Solvent, Soxhlet, Press	Amaral et al., 2003; Pereira et al., 2008; Bouabdallah et al., 2014; Rabadán et al., 2018d
Payne	Spain	2	8.02	7.92	8.11	0.13	2.87	2.49	3.25	0.54	21.92	17.59	26.25	6.12	55.51	52.81	58.20	3.81	10.92	9.29	12.55	2.31	Press	(Rabadán et al., 2018d)

(continued on next page)

Table 1 (continued)

Variety	Country	N	16:0				18:0				18:1ω9				18:2ω6				18:3ω3				Oil Ext. Mode	References
			Mean	Min	Max	Sdv	Mean	Min	Max	Sdv	Mean	Min	Max	Sdv	Mean	Min	Max	Sdv	Mean	Min	Max	Sdv		
Pedro	Spain, Turkey	5	6.64	5.97	7.25	0.62	2.90	2.38	3.97	0.73	15.94	14.47	21.39	3.05	61.04	58.25	62.13	1.59	12.92	9.11	15.14	2.49	Press, Soxhlet, Solvent	Kafkas et al., 2017; Rabadán et al., 2018a; Zhang et al., 2021
Quingxiang Rex	China New Zealand	2 3	6.39 7.26	5.55 6.59	7.23 7.70	0.59	1.06	2.08	3.87	0.87	13.71 13.59	13.57 12.66	13.84 14.30	0.84	67.15 61.99	65.43 61.20	68.87 62.48	0.69	9.39 15.26	9.22 14.20	9.55 16.17	0.99	Press Solvent, Press	Zhou et al., 2018; Zhang et al., 2021 Savage et al., 1999; Zwarts et al., 1999
R. Livermore	USA	1	8.03				2.27				18.34			61.69				9.46					Soxhlet	Kafkas et al., 2020
Sampion	Serbia	2	7.30	7.10	7.50	0.28	1.65	1.60	1.70	0.07	19.85	19.00	20.70	1.20	60.35	59.80	60.90	0.78	10.40	9.80	11.00	0.85	Press	Kafkas et al., 2011
Sebin	Turkey	3	7.29	6.84	7.81	0.49	2.65	2.58	2.71	0.09	20.53	19.06	21.45	1.29	55.17	55.00	55.49	0.27	13.10	12.49	14.17	0.93	Soxhlet	Beyhan et al., 2016; Yildiz and Karaca, 2021
Sejnovno	Turkey	2	6.50	6.30	6.70	0.28	1.70	1.70	1.70	0.00	16.05	15.90	16.20	0.21	64.20	63.30	65.10	1.27	10.90	10.60	11.20	0.42	Press	Rabrenovic et al., 2011
Sen	Turkey	1	6.58				3.23				26.86				53.24				9.50				Soxhlet	Kafkas et al., 2017
Serr	Argentina, Spain, Turkey	6	6.94	6.57	7.44	0.42	2.79	1.80	3.88	0.72	21.42	14.94	26.86	4.74	56.25	52.50	61.37	3.51	12.07	9.50	13.60	1.58	Soxhlet, Press	Martínez and Maestri, 2008; Tapia et al., 2013; Kafkas et al., 2017; Rabadán et al., 2018d
Solano	USA	1	9.47				9.27				10.85			61.50				15.65					Soxhlet	Kafkas et al., 2020
Sorrento	Argentina, Italy	3	8.01	7.16	8.65	0.77	3.07	1.71	3.75	1.17	13.79	11.95	17.20	2.96	58.98	58.90	59.03	0.07	15.99	15.00	16.85	0.93	Press, Soxhlet	Martínez and Maestri, 2008; Poggetti et al., 2018;
Stanley	New Zealand	3	6.94	6.70	7.40	0.40	1.29	0.08	2.00	1.06	19.72	18.80	20.36	0.82	59.65	59.10	60.60	0.83	11.56	11.18	12.20	0.56	Solvent, Press	Savage et al., 1999; Zwarts et al., 1999
Tehama	New Zealand, USA	3	7.40	6.80	7.80	0.53	1.65	1.35	2.00	0.33	19.08	18.10	19.60	0.85	59.33	57.88	60.40	1.30	12.19	11.10	13.10	1.01	Solvent, Press	Savage et al., 1999; Zwarts et al., 1999
Tiszacsécsi83	Hungary	2	6.60	6.37	6.82	0.32	2.33	2.19	2.47	0.20	17.36	16.18	18.53	1.66	60.79	59.18	62.39	2.27	10.73	10.46	10.99	0.37	Solvent	Bujdosó et al., 2010, 2016
Tongzigu	China	1	6.54				1.83				35.08			53.39				5.04					Press	Zhang et al., 2021
Toyserkan	Iran	1	8.81				3.09				25.13			50.15				12.04					Soxhlet	Gharibzahedi et al., 2014
Trompito	Argentina	1	5.78				2.30				18.22			60.39				13.32					Press	Cittadini et al., 2020
Tulare	Argentina	2	6.57	6.38	6.75	0.26	2.62	2.16	3.07	0.64	20.45	16.80	24.10	5.16	58.14	55.90	60.38	3.17	12.21	11.40	13.01	1.14	Press	Cittadini et al., 2020; Martínez and Maestri, 2008
Vina	France, New Zealand USA	4	5.74	2.40	7.30	2.25	1.53	0.60	2.20	0.70	15.71	7.00	19.90	5.88	61.78	58.00	72.00	6.83	14.79	12.70	17.60	2.11	Solvent, Press	Savage et al., 1999; Zwarts et al., 1999
Wen185	China	1	5.60				1.94				16.17			63.81				12.54					Press	Zhang et al., 2021
Xiangling	China	9	6.03	5.44	7.75	0.84	3.94	2.95	4.55	0.61	16.22	12.40	21.48	3.47	63.95	60.35	67.40	2.47	9.20	6.60	10.60	1.43	Soxhlet, Press	Li et al., 2017; Zhou et al., 2018; Gao et al. 2019a; Liu et al., 2020; Wei et al., 2020; Zhang et al., 2021
Yalova	Turkey	10	6.18	9.64	7.53	1.40	2.72	3.85	3.13	0.36	8.70	20.77	15.89	5.09	58.21	64.85	61.05	2.42	10.60	14.92	12.45	1.82	Soxhlet	Koyuncu et al., 2001
Yihe	China	1	5.42				2.23				17.47			65.17				9.46					Press	Zhang et al., 2021
Yonghe	China	3	5.56	5.00	5.93	0.49	2.83	2.61	3.12	0.26	27.74	18.32	37.39	9.54	54.58	44.22	63.52	9.73	8.58	7.63	9.10	0.83	Soxhlet	Li et al., 2017
Yunxin R3	China	1	5.53				2.31				22.51			62.18				7.24					Press	Gao et al., 2019c
Zha 343	China	1	5.41				2.45				31.96			51.23				7.87					Soxhlet	Li et al., 2017
Zhipi 3	China	1	5.26				3.09				23.19			58.04				7.64					Soxhlet	Li et al., 2017
Zhonglin	China	4	5.86	7.34	6.58	0.61	2.30	3.34	2.80	0.44	18.11	23.76	20.00	2.55	54.83	62.13	59.19	3.16	9.48	10.30	10.07	0.39	Soxhlet	Li et al., 2017
Zhonglin	China	4	5.86	7.34	6.58	0.61	2.30	3.34	2.80	0.44	18.11	23.76	20.00	2.55	54.83	62.13	59.19	3.16	9.48	10.30	10.07	0.39	Soxhlet	Li et al., 2017
Genotype	Iran	30	4.94	4.10	5.80	0.55	nd				16.13	11.20	21.95	3.10	50.67	44.00	61.00	4.80	15.06	11.20	21.10	2.60	Solvent	Akhiani et al., 2017
Genotype	Turkey	52	5.68	6.61	5.98	0.43	1.90	2.85	2.24	0.42	22.63	27.27	23.91	2.25	49.93	54.41	52.13	1.87	14.32	17.82	15.94	1.59	Soxhlet, Solvent	Dogan and Akgul., 2005; Ozkan and Koyuncu, 2005; Beyhan et al., 2016; Erturk et al., 2020 ;Ghafoor et al. 2020;; Ozcan et al., 2020
Genotype	China	19	5.92	3.05	8.25	1.16	2.60	1.00	5.59	0.91	18.70	12.56	26.03	3.39	62.53	51.21	68.97	4.81	9.66	6.83	15.01	2.09	Press, Soxhlet	Gao et al., 2018; Li et al., 2017
Genotype	Italy	70	3.90	11.37	7.73	1.16	1.40	5.23	2.75	0.88	10.05	25.09	16.00	2.97	56.01	68.62	61.05	2.73	6.89	16.18	12.16	2.10	Soxhlet	Poggetti et al., 2018
Genotype	New Zealand	2	7.19	6.65	7.73	0.76	1.73	1.40	2.05	0.46	18.02	16.46	19.58	2.21	59.54	57.09	61.98	3.46	12.58	12.45	12.71	0.18	Solvent	Savage et al., 1999
Genotype	Poland	7	7.50	6.60	8.20	0.20	2.60	2.00	3.10		18.90	17.20	20.40		58.50	58.30	60.70		11.70	10.10	12.50		Press	Grajzer et al. 2020
Genotype	USA	6	6.99	6.59	7.36	0.29	2.53	2.15	3.00	0.30	16.26	14.31	19.21	1.83	62.43	60.00	66.07	2.30	11.56	8.41	16.14	2.85	Soxhlet	Kafkas et al., 2020

N: sample number, Min: minimum value, Max: maximum value, Sdv: standard deviation, 16:0: palmitic acid, 18:0: stearic acid, 18:1ω9: oleic acid, 18:2ω6: linoleic acid, 18:3ω3: linolenic acid, Oil Ext. Mode : Oil extraction mode.

9. Triacylglycerols

Triacylglycerols (TG) are the major compounds in walnut oil. Their composition is correlated with that of fatty acids. The large number of fatty acids capable of esterifying glycerol generates many potential combinations, leading to complex mixtures of triacylglycerols. However, the alcohol functions of glycerol are not randomly esterified. Thus, position 2 of glycerol is preferably esterified with unsaturated fatty acids (oleic, linoleic and linolenic) (Crews et al., 2005).

The analysis of walnut oil triacylglycerols was performed by GC or High-Pressure Liquid Chromatography (HPLC) and did not require any sample pretreatment except filtration. Triacylglycerol separation by GC was carried out according to the total carbon number (IUPAC Method 2.210, 1992). Columns were short (15 m) and coated with non or mid polar stationary phases (methylpolysiloxane phases with different percentages of phenyl groups). The detector was a flame ionization detector (FID) (Crews et al., 2005; Bouabdallah et al., 2014; Gao et al., 2018, 2019c, b). The low volatility of triacylglycerols required high temperatures for the injector (360–380 °C), detector (375–380 °C) and oven (340–390 °C). Crew et al. (2005) used cooled on-column injection.

The composition of TGs separated by HPLC was determined by calculating their equivalent carbon number (ECN) ($ECN = CN - 2n$, where CN is the carbon number and n the number of double bonds in the triglyceride molecule) according to the standard method (IUPAC Method 2.210, 1992). In most of studies, the separation was performed at ambient temperature or at 40 °C with an octadecylsilane (C18) reversed stationary phase and an acetone/acetonitrile (70:30, v/v or 50/50, v/v) mixture as mobile phase. Detection was achieved using evaporative light-scattering or refractive index detectors (Oliveira et al., 2002; Bada et al., 2010). One study mentioned the use of two columns NovaPak C18 (300 × 3.9 mm + 150 × 3.9 mm, 4 μm, 25 °C) connected in series, with mobile phase gradient of 2-propanol in acetonitrile, and UV (205 nm) detector, evaporative light-scattering detector and positive-ion APCI-MS connected in series. For each of the three detection techniques, the quantification of triacylglycerols was carried out by considering their response factors (Holčapek et al., 2005). Ayorinde et al. (1999) analysed TGs using Matrix-assisted Laser Desorption/Ionization Time-of-flight Mass Spectrometry (MALDITOF). A-Cyano-4-hydroxycinnamic acid, dissolved in acetonitrile/tetrahydrofuran, was used as the matrix. The MALDITOF mass spectra produced only sodiated triacylglycerol molecules $[M + Na^+]$ (Ayorinde et al., 1999).

Tables 2 and 3 list TG contents in walnut oils calculated from data reported in the literature (mean, minimum and maximum), according to the variety, the country, the oil extraction process and the authors. The main TGs (i. e., LLLn (5.59–23.46%), LLL (23.70–44.26%), OLLn (0.83–32.38%), OLL (6.40–27.57%) and PLL (4.83–17.59%), with P: palmitoyl, O: oleoyl, L: linoleoyl and Ln: linolenoyl) contained at least one linoleoyl chain, which showed the correlation with the high percentages of linoleic acid (50.15 to 74.00%) found in oils. Authors also found TGs, namely OOL, PLO and PLLn, in lower percentages (around 2% and 5%) and LLnLn and POO less than 2%. Some other minor TGs often detected in other vegetable oils (especially OOO, SOS, SLL and SOL, with a stearyl chain (S)) have been quantified (Bada et al., 2010; Bouabdallah et al., 2014; Gao et al., 2018, 2019b, c; Oliveira et al., 2002). In addition, TG contents showed significant differences. However, the few articles dealing with the TG content in walnut oils did not show any correlation with any parameter (variety, geographic origin, extraction process or analytical method used).

Some authors compared the influence of different oil extraction processes from the kernels. No difference in TG compositions was found between hexane extracted oils by Soxhlet apparatus and SC-CO₂ extracted oils (Oliveira et al., 2002). Gao et al. (2019a) compared the TG compositions in oils obtained by extracting walnut kernels of the Xiangling variety with five solvents (hexane, ethyl acetate, petroleum ether, acetone, and chloroform / methanol (2/1, v/v)), cold pressing being taken as a reference. The LLL content of the cold pressed oil

(38.54%) was significantly higher than that of the solvent extracted oils. No significant difference was observed in POL, SOS, OLLn, and LLLn contents whatever the solvent used for the oil extraction (Gao et al., 2019b).

The fine and complete identification of TGs is often a complex challenge because of low resolution and many overlapping chromatographic peaks, which explains why researchers rather analysed the FAs.

10. Tocopherols and tocotrienols

Tocopherols (T) and tocotrienols (T3), grouped under the name "vitamin E", are important fat-soluble compounds in vegetable oils. Both come in four forms depending on the degree of methylation of the chromanol ring, with the α tocopherol being trimethylated, the β tocopherol (positions 5 and 8) and γ tocopherol (positions 7 and 8) being dimethylated, and the δ tocopherol (position 8) being monomethylated. Tocopherols are major fat-soluble vitamin and thanks to their hydrogen-donating power act as potential antioxidant during unsaturated lipid oxidation via chain-breaking reactions. α-tocopherol and δ-tocopherol seems having higher antioxidant potency than γ-tocopherol (like γ-tocopherol) (Yang et al., 2018). Therefore, they are widely studied in walnut oils by researchers seeking to highlight their nutritional potential.

Tocopherols were almost systematically analysed by high performance liquid chromatography (HPLC) from a simple dilution of the oil sample in the appropriate solvent. However, several authors carried out an oil saponification procedure (KOH solution in methanol or ethanol) at a temperature of 70–90 °C followed by solvent extraction (hexane, hexane / ethyl acetate or methanol) before HPLC injection (Maguire et al., 2004; Miraliakbari and Shahidi, 2008a; Nosenko et al., 2019; Pycia et al., 2019a, b). Some studies (Derewiaka et al., 2014; Gharibzahedi et al., 2014; Bujdosó et al., 2016; Beyhan et al., 2017) referred to the experimental procedure developed by Maguire et al. (2004). The normal phase (NP) polarity mode was the most used: the column (250 mm × 4.6 mm × 5 μm in most studies) was filled with silica gel as stationary phase, the mobile phase consisting of a very low percentage of isopropanol (from 0.2% to 2%) in hexane for the optimal separation of all the homologues (Oliveira et al., 2002; Amaral et al., 2005; Martínez et al., 2006; Li et al., 2007; Miraliakbari and Shahidi, 2008a; Schwartz et al., 2008; Bada et al., 2010; Czaplicki et al., 2011; Abdallah et al., 2015; Rueda et al., 2016; Zhou et al., 2016, 2018; Gao et al., 2018, 2019c, 2021; Ojeda-Amador et al., 2018; Rabadán et al., 2018d; Ahmed et al., 2019; Bakkalbaşı, 2019; González-Gómez et al., 2019; Cittadini et al., 2020). Heptane was sometimes substituted for hexane (safer for human health) (Schwartz et al., 2008, Ahmed et al., 2019) and isopropanol was replaced by either tert-butylmethylether (Li et al., 2007; Ahmed et al., 2019) or 1,4-dioxane (Amaral et al., 2005; Schwartz et al., 2008), or even ethanol (Bujdosó et al., 2010). A few authors used a NH₂ grafted stationary phase, with either a heptane / tert-butylmethylether / tetrahydrofuran / methanol mixture (79/20/0.98/0.02, v/v) (Savage et al., 1999) or a hexane / ethyl acetate mixture (70/30, v/v) (Cittadini et al., 2020) as mobile phase, or a diol grafted stationary phase with heptane as mobile phase (Ghafoor et al., 2020) or a hexane / tert-butylmethylether mixture (96/4, v/v) (Crews et al., 2005; Matthäus et al., 2018). Other authors performed the analysis of tocopherols in reversed phase (RP) polarity mode using a C18 stationary phase (Lavedrine et al., 1997; Maguire et al., 2004; Kornsteiner et al., 2006; Rabrenovic et al., 2011; Gharibzahedi et al., 2013, 2014; Zhai et al., 2015; Beyhan et al., 2016, 2017; Iqbal et al., 2016; Kodad et al., 2016; Kafkas et al., 2017; Pycia et al., 2019a, d; Grajzer et al., 2020; Özcan et al., 2020; Wu et al., 2020; Grilo and Wang, 2021; Laskos et al., 2021; Yildiz and Karaca, 2021) or an octylsilane (C8) stationary phase (Derewiaka et al., 2014; Nosenko et al., 2019). The mobile phases most often used in isocratic mode consisted of 0 to 10% of water in methanol. However, some authors used different percentages of solvents in methanol: dichloromethane (15%, (Kafkas et al., 2017)) or acetonitrile (30%, (Kodad et al.,

2016); 50%, (Iqbal et al., 2016)), methanol/acetonitrile/water/ethyl acetate (Laskos et al., 2021) or methanol / water / methylterbutylether mixture (Grajzer et al., 2020) used in gradient mode. One study mentioned the use of acetonitrile in an aqueous mobile phase (Nosenko et al., 2019). A hexane / 2-propanol isocratic mobile phase (99.5/0.5, v/v or 98/2 v/v) was also used (Gharibzahedi et al., 2013, 2013; Beyhan et al., 2016; Wu et al., 2020) even if it does not seem to be compatible with the C18 stationary phase. In both polarity modes, a UV detector (λ from 209 to 298 nm, most often $\lambda = 295$ nm) or a fluorescence detector (mainly $\lambda_{ex}=290$ or 295 nm and $\lambda_{em}=330$ or 320 nm) was used. A method of identification by comparison of authentic standard retention times was indicated in most papers. In some studies (Zhai et al., 2015; Kornsteiner et al., 2006; Martínez et al., 2006) when detection was carried out using a UV detector, only the γ - and δ -T homologues were detected. This may be explained by the lower sensitivity of the UV detector compared with that of the fluorescence detector. Some authors have used MS detection (Laskos et al., 2021).

The NP-HPLC assay method allowed the separation, detection and quantification of the four tocopherol homologues and the four tocotrienol homologues eluted in the following order: α -T, α -T3, β -T, γ -T, β -T3, γ -T3, δ -T and δ -T3. Tocotrienols were quantified only by Amaral et al. (2005) and Schwartz et al. (2008) on a silica column, and by Crews et al. (2005) and Matthäus et al. (2018) on a diol grafted stationary phase. By contrast, the RP- HPLC assay method did not allow the separation of β -T and γ -T. This inefficiency was mentioned by Lavedrine et al. (1997), who claimed that this was not a problem since the absence of β -T in walnut oil had been shown. Unfortunately, the authors did not comment on a possible co-elution. It should be noted that studies following those of Lavedrine et al. (1997), carried out in normal phase, revealed small amounts of β -T in walnut oil (references mentioned above). β -T and γ -T were quantified together by certain authors (Kornsteiner et al., 2006; Rabrenovic et al., 2011; Beyhan et al., 2016; Kafkas et al., 2017) since β -T was not resolved from γ -T by RP-chromatography, as specified by Kornsteiner et al. (2006) and Beyhan et al. (2016). In contrary with these studies, it was surprising that the four tocopherols could be separated by Wu et al. (2020) who used a RP-C18 stationary phase, with a mobile phase generally used with a normal phase. In other studies, β -T was not mentioned and only α -T, γ -T and δ -T were quantified (Zhai et al., 2015; Kodad et al., 2016). Maguire et al. (2004) quantified only α -T and γ -T. Some studies mentioned different results: (i) β -T was co-eluted with δ -T with a 3% water in methanol or 100% methanol mobile phase on a RP-C18 stationary phase (Beyhan et al., 2017; Pycia et al., 2019a, 2019b), (ii) β -T, identified alongside α -T and the sum γ -T + δ -T with a monomeric C8 stationary phase and an acetonitrile / water mobile phase (70/80, v/v) was found as the major tocopherol (Nosenko et al., 2019). The composition of the tocopherol homologues was also determined by GC on a silica capillary column coated with 35% dimethyl-65% diphenyl-polysiloxane, with helium as vector gas. γ -T and δ -T were successfully quantified (Verardo et al., 2009). The total tocopherol content was quantified by a spectrophotometric method in which tocopherols and tocotrienols reduced ferric ions to ferrous ions, which reacted to form a coloured complex with dipyrindyl (absorbance at 510 nm) (Martínez and Maestri, 2008; Martínez et al., 2011; Nosenko et al., 2019). Authors claimed that this simpler and cheaper method was sufficient to afford a global determination of tocopherols and was suitable for routine use in quality control laboratories.

Table 4 presents the mean, minimum and maximum content of each tocopherol, calculated from data reported in the literature, according to the walnut variety. There are noticeable variations in tocopherol contents, particularly for γ -T (Nguyen and Vu, 2021). As Martínez et al. (2010) already mentioned, “ γ -T was the major tocopherol, followed by δ - and α -T. β -tocopherol was not detected in most cases”. γ -T represented around 85% of the total tocopherols and reached values between 97 and 677 mg/kg with an average of 281 mg/kg oil. The δ -T content represented around 10% of the total tocopherols and reached an average

value of 31 mg/kg oil (from 8 to 107 mg/kg), the α -T content reached an average value of 18 mg/kg oil (5%, from 0 to 144 mg/kg), and the β -T content reached a maximum value of 28 mg/kg oil (average of 1 mg/kg, less than 1%). As for tocotrienols, the few authors that mentioned them reported an average content of γ -T3 around 3 mg/kg (from 0.71 to 5.14 mg/kg), and only one value of 1.99 mg/kg of δ -T3 was reported by Matthäus et al. (2018). In the other studies, this compound and the other two homologues were only found in trace amounts.

Tocopherol contents were much more strongly influenced by the crop years than by the varieties (Martínez et al., 2006), for which only minor differences were observed (Wu et al., 2020). Growing conditions, harvest time or the geographic area of growing can be sources of variation of tocopherol contents (Nguyen and Vu, 2021). The γ -T content was influenced by neither the levels of nitrogen fertilisation nor the amount of irrigation (Verardo et al., 2009). The harvest period affected the tocopherol contents of walnut oils. The earlier the harvest, the higher the tocopherol contents, but the lower the oil yield (Matthäus et al., 2018). The tocopherol contents increased with the maturation of the walnuts (Pycia et al., 2019a). The geographical location seems to impact the tocopherol content since, for walnut samples from the main cultivars of growing areas in China, the total tocopherol, γ -T and δ -T average contents decreased from the northwest zone to the east zone, and to the southwest zone (Gao et al., 2018).

Some authors studied the stability of tocopherols during the storage time of either the oil or the walnuts before the oil extraction, or even during the treatment of walnuts against food-borne pathogens (i.e., gamma irradiation). The tocopherol content remained unchanged up to 4 kGy and significantly declined above (Iqbal et al., 2016). A microwave pre-treatment of walnut kernels (implemented to improve the flavour and oxidative stability of walnut oil) led to a tocopherol content decrease, increasing along with the treatment duration (Zhou et al., 2016). Roasting process did not have significant effect on tocopherols contents of oils extracted from different walnut varieties (Ghafoor et al., 2020). After a conservation of walnut kernels in light, temperature- and humidity-controlled chambers over 28 weeks, tocopherol content in oil extracted decreased by 20–23% depending on the variety (Grilo and Wang, 2021). A study investigated on the effect of the walnut skin darkening (occurring during the pre- and post-harvest stages) and the storage conditions of walnuts. Lower α - and γ -tocopherol contents have been found in oil extracted from the dark-skin walnuts than from the light-skin walnut oil. Over the 6-month storage, tocopherol contents decreased more intensely for walnuts in the packages with air and at non-refrigerated conditions than for walnuts in oxygen-free packages (i.e., vacuum or N_2 atmosphere) and refrigerated. Nonetheless, both types of ground walnuts were acceptable according to the oxidation parameter (Yildiz and Karaca, 2021).

The γ -T content was higher in cold-press extracted oils than in Soxhlet extracted oil using petroleum ether (Ahmed et al., 2019). Cold chloroform / methanol extraction (1/1, v/v) gave oils with higher tocopherol contents than cold hexane extraction, without however modifying the homologue relative composition (Miraliakbari and Shahidi, 2008b). Chloroform / methanol mixture (2/1, v/v) was the most suitable for the extraction of tocopherols, compared with hexane, ethyl acetate, petroleum ether or acetone (Gao et al., 2019b). A higher tocopherol contents were obtained by cold extraction with water / methanol / chloroform mixture than by maceration or cold-press extraction (Gharibzahedi et al., 2013). Tocopherol contents have been found significantly lower in SC-CO₂ extracted oil than in cold pressing oil (Crowe and White, 2003). Conversely, a SC-CO₂ extracted oil (70 °C, 200 bar) presented larger tocopherol content than cold pressing oil. SC-CO₂ extracted oil presented also larger total amount of tocopherols compared with hexane-extracted oil by using a Soxhlet apparatus, with higher α -T, lower δ -T and unchanged γ -T percentages (Oliveira et al., 2002). A recent study showed that a SC-CO₂ extracted oil had the highest γ -T content while SBE extracted oil had the higher δ -T and total tocopherol contents. However, the total tocopherol content in a SC-CO₂

extracted oil was the same than in cold pressing oil (Gao et al., 2021). Tocopherol contents in walnut oil extracted by enzyme-assisted aqueous methodologies were higher than in the oil extracted with hexane (González-Gómez et al., 2019).

Furthermore, α -T, experimentally added to oil, has been shown to inhibit the formation of hexanal (a secondary oxidation product) (Bakkalbaşı, 2019). In cold pressed walnut oils in which herbs have macerated (common basil, sage, and thyme) the tocopherol level did not play a decisive role in determining the antioxidant properties of oils and only the addition of 50 g / 100 ml of oil of basil leaves has induced an increase in the tocopherol content (Laskos et al., 2021).

11. Sterols, methyl sterols, triterpenic and aliphatic alcohols

Triterpenic alcohols (4,4-dimethylsterols), methylsterols (4-monomethylsterols) and sterols (4-demethylsterols) constitute the triterpene pathway whose origin is squalene and the end step, sterols. These compounds are characteristic of the authenticity of vegetable oils and are compounds of interest for the control of vegetable oil purity and origin. They are constituents from the unsaponifiable matter and are usually analysed in the same way.

Analytical protocols are often derived from a standardised method NP EN ISO 12228 (Amaral et al., 2003; Zhou et al., 2016) or EEC Regulation 2568/91 established for olive oil (Oliveira et al., 2002; Rabadán et al., 2018a, c, d). First, an oil saponification was required (KOH or NaOH/EtOH at various concentrations). Four studies analysed the sterol fraction without any mentioned purification (Martínez et al., 2006; Czaplicki et al., 2011; Derewiaka et al., 2014; Grajzer et al., 2020). In the other studies, the unsaponifiable matter were fractionated/purified on an alumina oxide column and/or on a silica thin layer. Matthäus et al. (2018) and Martínez et al. (2006) performed a chromatographic analysis without prior derivatisation. The other authors have used different silylation reagents for a conversion into trimethyl silyl derivatives: hexamethyldisilazane/trimethyl chlorosilane/pyridine (Oliveira et al., 2002; Verardo et al., 2009; Bada et al., 2010), N-methyl-N-(trimethylsilyl) trifluoroacetamide / 2-mercaptoethanol / NH_4I (Abdallah et al., 2015), N-methyl imidazole + N-methyl-N-(trimethylsilyl)-heptafluorobutyramide (Amaral et al., 2003), N, O-bis (trimethylsilyl) trifluoroacetamide with 1% trimethyl chlorosilane. The obtained derivatives were further analysed by GC on 25–60 m long columns, with a widely used phase composition of 5% phenyl and 95% polydimethylsiloxane. Qualitative analysis was performed by comparing the retention times with those of commercial reference compounds (β -sitosterol, campesterol and stigmasterol) or sterols from oils of known composition and their mass spectra. Quantitative analysis was performed by adding an internal standard, such as 5 α -cholestane, 5 α -cholestan-3 β -ol, betulin and dihydrocholesterol, during saponification. Maguire et al. (2004) were the only ones to analyse free sterols by HPLC using a C8 column with an acetonitrile/water mobile phase (80/20, v/v) and an UV detection at 205 nm.

Some authors presented major and minor sterol compositions (Amaral et al., 2003; Crews et al., 2005; Bada et al., 2010; Martínez et al., 2010; Abdallah et al., 2015; Grajzer et al., 2020). The three most mentioned sterols were β -sitosterol (ultra-majority), Δ 5-avenasterol and campesterol. The campesterol content was always found to be higher than the stigmasterol content, as is usual in most vegetable oils, except in two studies (Maguire et al., 2004; Czaplicki et al., 2011). In most studies, the β -sitosterol content was found above 80% of the sterol fraction. It ranged from 751 mg/kg oil (Yang et al., 2018) to 2250 mg/kg oil (Crews et al., 2005) from one variety to another. The Δ 5-avenasterol content ranged from 25 mg/kg oil (Amaral et al., 2003) to 153 mg/kg oil (Crews et al., 2005) between varieties. The campesterol content ranged from 4.95 mg/kg oil (Abdallah et al., 2015) to 121 mg/kg oil (Crews et al., 2005) between varieties. In some studies, the apparent β -sitosterol content (i.e. the sum of several sterols: β -sitosterol, sitostanol, Δ 5, 24-stigmastadienol, clerosterol and Δ 5-avenasterol contents) was given

in addition to the β -sitosterol content (Abdallah et al., 2015; Sena-Moreno et al., 2016; Rabadán et al., 2018d). According to Grajzer et al. (2020), Δ 7-avenasterol was found in quantities at least 10 times greater than those published by other authors, perhaps because of a lack of purification of unsaponifiable fraction according to the described method.

In oils from six varieties of walnuts, the total triterpenic alcohol content ranged from 242 to 620.08 mg/kg oil, including cycloartenol as major compound (226.45 to 532.06 mg/kg oil), butyrospermol (7.51 to 86.56 mg/kg) and 24-methylenecycloartenol (3.54 to 52.2 mg/kg) (Abdallah et al., 2015). In another study, cycloartenol (10 mg/kg oil) was the only triterpenic alcohol quantified (Czaplicki et al., 2011). These results confirmed previous quantification of cycloartenol and 24-methylenecycloartenol (62.62% to 85.94%, and 5.29 to 26.78% of the triterpenic alcohol fraction, respectively) in the oils of three varieties of walnuts harvested over two successive years (Martínez et al., 2006). Two methyl sterols were identified: cycloeucaenol (Martínez et al., 2006) and citrostadienol (Czaplicki et al., 2011). Hexacosanol was the dominant aliphatic alcohol (9.70 –28.14 mg/kg oil) followed by tetracosanol (4.14 –12.71 mg/kg oil), and octacosanol and docosanol were present in minor quantities (Abdallah et al., 2015). A great heterogeneity was observed in literature results, not only on the compounds identified but also on their content. This may be due to the origin of the oil samples but also to differences in sample preparation and analysis. Further studies would be necessary to clarify the triterpenic alcohol composition of walnut oils.

A microwave pre-treatment of kernels led to a decrease in the sterol content proportional to the treatment duration (Zhou et al., 2016). Sterols were affected by the agricultural practices such as irrigation and nitrogen fertilisation. The highest levels of total sterols were obtained for low levels of nitrogen (60 kg/ha/an) and copious amounts of water (100% calculated on daily evapotranspiration) (Verardo et al., 2009). Total sterol contents varied according to the harvest times and evolved from 1460 to 4847 mg/kg when walnut fruits were harvested at ten days intervals (five harvest periods) in Antalya province in Turkey (Matthäus et al., 2018). Nguyen and Vu (2021) have compiled phytosterol content of *Juglans reggia* from different works in which origin geographic has a weak impact on phytosterol composition. Amaral et al. (2003) claimed that phytosterol content was a good tool to access authenticity of variety. Several authors investigated whether oil extraction techniques influenced the sterol content. No significant differences in the sterol composition were found between oils obtained either by SC-CO₂ extraction or by hexane Soxhlet extraction (Oliveira et al., 2002) nor between oils obtained using either a hydraulic press or a screw press (Sena-Moreno et al., 2016). The temperature in the screw press did not affect the oil sterol composition (Rabadán et al., 2018c). Additionally, the total sterol amounts in cold-pressed walnut oils were not modified by variable storage temperature conditions (from 5 °C to 20 °C) after 16-month storage (Rabadán et al., 2018a).

12. Phenolic compounds

Phenolic compounds in walnut oils were quantified through the total phenolic compounds (TP) and ortho-diphenolic compounds (OD) (Table 5). However, some data on the individual phenolic compounds were communicated. The TP content in oil samples was determined according to different methods using the Folin-Ciocalteu reagent on phenolic compounds extracted from walnut oil with methanol or methanol / water mixtures. TP content was quantified by visible absorptiometry from a calibration curve and expressed in standard equivalent. The use of different standards (caffeic acid (CAE), gallic acid (GAE), and D-catechin) made their comparison difficult. Moreover, a likely interference of phospholipids with phenolic compounds quantified by spectrophotometric measurements was mentioned but could not be established because of a lack of knowledge of their exact contributions (Arranz et al., 2008). There were very few studies on the OD in walnut oils. For their analyse, in methanolic extracts, an ethanolic / water solution of

Table 2
Relative percentages of triacylglycerols (LLnLn, LLLn, LLL, OLLn, PLLn) for the studied cultivars.

Variety	Country	N	LLnLn				LLLn				LLL				OLLn				PLLn				Oil Ext. Mode	References
			Mean	Min	Max	Sdv	Mean	Min	Max	Sdv	Mean	Min	Max	Sdv	Mean	Min	Max	Sdv	Mean	Min	Max	Sdv		
Arco	Portugal	1	2.60				22.64				36.70				5.33				2.86				Solvent	Amaral et al., 2004
Chandler	China	1	nd				5.95				33.94				21.06				nd				Press	Gao et al., 2019c
Franquette	Portugal, Tunisia	5	1.64	0.99	2.12	0.54	16.86	11.72	19.99	3.21	34.79	30.28	40.43	3.71	4.53	1.03	5.81	2.01	2.67	1.46	3.59	0.9	Solvent	Amaral et al., 2004; Bouabdallah et al., 2014
Hartley	China, Portugal, Spain, Tunisia	4	1.14	0.35	1.93	1.12	12.82	8.26	19.77	6.12	35.74	33.01	41.04	3.60	8.91	0.83	19.83	9.81	2.66	2.60	2.71	0.08	Solvent, Press	Amaral et al., 2004; Bouabdallah et al., 2014; Rabadán et al., 2018b; Gao et al., 2019c;
Hugget	Spain	1									33.96												Press	Rabadán et al., 2018d
Lara	Portugal	4	1.86	1.00	2.56	0.65	21.09	19.36	22.74	1.41	40.40	39.15	41.38	0.96	4.38	3.69	4.72	0.48	2.08	1.30	2.70	0.58	Solvent	Amaral et al., 2004
Lauzerone	Tunisia	1	0.53				7.15				30.00				0.44				2.24				Unknown	Bouabdallah et al., 2014
Marbot	Portugal	2	1.46	0.82	1.81	0.55	17.34	12.67	20.49	4.12	38.42	37.79	39.00	0.61	4.63	3.87	5.59	0.88	2.02	1.28	2.4	0.64	Solvent	Amaral et al., 2004
Mayette	Portugal, Spain	5	2.03	0.98	2.69	0.75	19.52	13.65	23.46	4.16	36.18	30.41	39.24	3.48	4.86	4.23	5.39	0.55	2.56	1.09	3.47	1.03	Solvent, Press	Amaral et al., 2004; Rabadán et al., 2018d
Mellanaise	Portugal	4	1.26	0.86	1.8	0.44	16.07	13.64	18.14	2.04	37.71	35.83	39.62	1.66	4.76	4.03	5.65	0.67	1.88	1.44	2.54	0.52	Solvent	Amaral et al., 2004
Nerpio	Spain	1	nd				nd				35.22				nd				nd				Press	Rabadán et al., 2018d
Parisienne	Portugal, Spain, Tunisia	6	1.22	0.61	1.65	0.43	15.02	12.10	18.71	2.58	34.66	30.15	44.26	5.22	4.20	1.26	5.64	1.80	2.08	0.71	4.05	1.24	Solvent, Press	Amaral et al., 2004; Bouabdallah et al., 2014; Rabadán et al., 2018d
Payne	Spain	1	nd				nd				34.14				nd				nd				Press	Rabadán et al., 2018d
Pedro	Spain	1	nd				nd				32.75				nd				nd				Press	Rabadán et al., 2018d
Rego	Portugal	1	2.71				23.11				23.87				5.35				1.96				Solvent	Amaral et al., 2004
Scharch-Franquette	Spain	1	nd				nd				24.34				nd				nd				Press	Rabadán et al., 2018b
Serr	Spain	1	nd				nd				30.18				nd				nd				Press	Rabadán et al., 2018d
Xiangling	China	6	nd				6.59	5.58	8.37	0.97	34.84	32.06	38.54	2.55	20.32	6.68	32.38	8.17	nd				Press	Gao et al., 2019a
Yunxin	China	1	nd				6.66				32.57				25.03				nd				Press	Gao et al., 2019c
E cvs	China	5	nd				10.71	8.23	12.43	1.84	35.44	31.28	39.47	3.09	20.94	18.16	24.18	2.5	nd				Press	Gao et al., 2018
NW cvs	China	5	nd				9.67	7.17	11.03	1.76	30.12	26.44	33.29	3.37	18.75	17.07	19.82	1.19	nd				Press	Gao et al., 2018
SW cvs	China	5	nd				8.12	6.42	10.06	1.64	31.68	27.87	35.13	2.92	21.92	20.44	23.51	1.27	nd				Press	Gao et al., 2018
Unknown	Portugal, Spain	6	3.58	2.85	4.40	0.63	16.42	14.63	18.40	1.55	25.98	23.70	29.20	1.92	5.70	4.60	7.30	1.04	4.23	3.30	5.18	0.73	Press	Oliveira et al., 2002; Bada et al., 2010

L: linoleoyl; Ln: linolenoyl; O: oleoyl; P: palmitoyl; Mean: mean value or value when single sample; Min: minimum value; Max: maximum value; N: sample number; Sdv: standard deviation; nd: not detected; Oil Ext. Mode: Oil extraction mode; E cvs: Eastern cultivars; NW cvs: Northwestern cultivars; SW cvs: Southwestern cultivars

sodium molybdate dihydrate was added. catechol, hydroxytyrosol and hydroxytyrosyl acetate were used to establish the calibration curve and results were expressed in mM o-diphenols per kg of oil (Arranz et al., 2008; Gharibzadeh et al., 2013, 2014). The individual phenolic compounds were extracted from walnut oil with methanol / water in different proportions and analysed by HPLC or by UHPLC on an octadecyl silane stationary phase with methanol / water + 1% phosphoric acid or water + 0.1% formic acid / acetonitrile + 0.1% formic acid or acetonitrile / methanol / water + formic acid mixtures used as mobile phase in gradient mode. Detection was performed with a UV or diode detector (280 nm and 350 nm) alone or associated with a MS or ESI/MS/MS detector (Slatnar et al., 2015; Rueda et al., 2016; Ojeda-Amador et al., 2018). TP content expressed in GAE equivalent, the most widely used standard, ranged from 4.1 to 468 mg GAE/kg oil depending on the walnut varieties. The OD content ranged from 0.20 to 0.37 mM o-diphenols/kg oil. Among the identified phenols, quercetin was found to be the main compound (7.19 mg/kg oil). Tyrosol (3.49 mg/kg oil), o-vanillin (1.27 mg/kg oil), pinoresinol (2.48 mg/kg oil) and seven phenolic acids (7.73 mg/kg oil for sum of them) were also detected (Rueda et al., 2016). Glansreginin A (0.025 mg/kg oil) and Glansreginin B (0.035 mg/kg oil), which belong to the hydrolysable tannin family (i.e., dicarboxylic acid derivatives) were also identified (Slatnar et al., 2015). The presence of these two compounds was confirmed in three cultivar oils: Glansreginin A (0.37 - 0.85 mg/kg oil) and Glansreginin B (0.03 - 0.17 g/kg oil) (Ojeda-Amador et al., 2018). Two other phenolic compounds were identified: di-hexahydroxydiphenoyl-glucose and di-hexahydroxydiphenoyl-galloyl glucose 1. Several peaks, which generally accounted for > 50% of the total content, could not be identified (Ojeda-Amador et al., 2018).

When oil was extracted from walnuts having submitted the same agricultural practices and the same extraction process, significant differences on individual phenolic compounds and TP contents of walnut oil have been determined among the analyzed cultivars (Gharibzadeh et al., 2014; Slatnar et al., 2015; Gao et al., 2019c). One author tried to show a significant impact of harvest year on TP content of nine walnut varieties which ranged from 9.53 mg to 40.93 mg CAE/kg oil. TP content varied systematically from one year to another (Rabadán et al., 2018d). Few studies analyzed the changes in the content of phenolic compounds according to the fruit maturity and oil storage conditions. Amin et al. (2017) reported that TP content decreased from 80.56 to 65.83 (mg GAE/g extract) during the walnut maturity. After 16 month of storage, TP content decreased slightly under different temperature and light expositions (Rabadán et al., 2018a). The influence of the walnut oil extraction process on the TP content was also studied. When cold solvent extraction was performed, acetone led to the highest TP content (98.1 mg GAE/kg oil), petroleum ether to the lowest (26.3 mg GAE/kg oil), and chloroform / methanol (2/1, v/v), hexane and ethyl acetate to intermediate contents (Gao et al., 2019b). In a study investigating on the effects of five oil extraction processes, the highest TP content (45.4 mg GAE/kg oil) was obtained by cold hexane extraction and the lowest content by SC-CO₂ extraction (28.0 mg GAE/kg oil). Cold pressing, roast pressing and SBE led to intermediate contents (Gao et al., 2021). Two aqueous enzyme-assisted methods (favouring either the highest oil yield or the lowest oxidative stability index) for oil extraction of walnut kernels were optimised using a response surface methodology. One of the two optimised methods led to a higher TP (74.2 mg CAE/kg oil) than the hexane extraction chosen as reference method (68.4 mg CAE/kg oil) (González-Gómez et al., 2019).

13. Hydrocarbons and squalene

Non-volatile hydrocarbons are minor compounds found in the unsaponifiable matter of edible oils. They are linear saturated (n-alkanes), branched saturated or unsaturated hydrocarbons. Squalene (2,6,10,15,19,23-hexamethyl-2,6,10,14,18,22-tetracosahexaene) is the main of them. The endogenous origin of n-alkanes in vegetable oils has been established from decarboxylation of long-chain fatty acids (Moreda et al., 2001). Nevertheless, they could also derive from mineral oil

contamination during the harvest, the conditioning, the oil extraction process, the transportation, or the packaging materials (Brühl, 2016). There are few data in the literature on the natural hydrocarbon content of walnut oil. Mainly, n-alkanes and squalene were mentioned in the unsaponifiable matter of walnut oil. McGill et al. (1993) studied the n-alkane compositions of twenty-one edible oils from the retail market, including three walnut oils. Walnut oils were fractionated on a silicic acid column, then purified by HPLC and analysed by GC and GC/MS on a nonpolar 100% dimethylpolysiloxane phase. Odd n-alkanes, namely n-nonadecane (C29) and n-hentriacontane (C31), were the main hydrocarbons, and their total content ranged from 7 to 30 mg/kg oil.

In other study (Martínez et al., 2006), the hydrocarbon composition was determined by GC and GC/MS on a low polar 5% phenyl - methylpolysiloxane stationary phase after saponification of the oil from three varieties of walnuts, from two harvest years. The results were expressed in % of the hydrocarbon fraction (HF). The main alkanes were even alkanes: tetradecane (C14, 6.70 to 13.98% HF), hexadecane (C16, 17.54 to 22.98% HF), octadecane (C18, 21.89 to 34.66% HF) and eicosane (C20, 11.36 to 14.40% HF), and were associated with small amounts of branched alkanes and alkenes (Martínez et al., 2006). In another study, the odd n-alkanes were predominant (McGill et al., 1993). According to Martínez et al. (2006), differences between their results and those obtained by McGill et al. (1993) could be attributed to genetic and/or environmental factors. However, the hydrocarbon composition of edible oils was widely studied, and the predominance of long chains of odd-numbered carbon atoms was well documented (Moreda et al., 2001). A complementary study should be necessary to confirm or to refute the divergent results of the two authors for walnut oils.

Squalene is a naturally occurring terpenoid hydrocarbon in the unsaponifiable matter of numerous edible vegetable oils, especially olive and pumpkin oil (Moreda et al., 2001). Squalene was considered as a scavenger of peroxy radicals inhibiting oxidation of both n-3 and n-6 fatty acids with high efficacy (Grajzer et al., 2020). But there were few data in the literature on the squalene content in walnut oil. Squalene was generally analysed from the unsaponifiable matter of walnut oil (Maguire et al., 2004; Derewiaka et al., 2014; Gao et al., 2018, 2019c, b; Cittadini et al., 2020). Czaplicki et al. (2011) analysed it directly from oil diluted in hexane. The squalene quantification was performed by GC on a 5%-Phenyl-methylpolysiloxane stationary phase or equivalent (VP-5 ms, DB-5) using FID or MS for detection and 5 α -cholestane or squalane as internal standard for quantification (Gao et al., 2018, 2019c; Cittadini et al., 2020). Several authors analysed the squalene content of a single sample of walnut oil (which was compared to other edible oils) by reverse phase HPLC of C18 or C8 using a UV detector at 215 nm or a DAD detector at 215 or 218 nm (Maguire et al., 2004; Czaplicki et al., 2011; Derewiaka et al., 2014).

Maguire et al. (2004) found a content of 9.4 mg/kg oil while Derewiaka et al. (2014) did not detect squalene. Czaplicki et al. (2011) gave an extremely high value of 28.3 mg/g oil compared with known levels in the literature, but in their work the squalene content value was expressed in non-homogeneous units with respect to other studied compounds. Squalene contents ranged from 17.97 to 131.45 mg/kg oil, with a mean value of 57.9 mg/kg oil for sixteen walnut oil samples of unspecified varietal origin but from three different regions of China with significantly different average values for the northwestern region (44.40 mg/kg), the eastern region (50.22 mg/kg) and the southwestern region (64.13 mg/kg) (Gao et al., 2018). The squalene content around 10-fold lower has been observed for three varieties (harvested in the same location in Yunnan Province, China) with low significant difference between varieties (from 4.41 to 5.21 mg/kg oil) (Gao et al., 2019c). Seven oils coming from manufacturers in Poland have shown a very different squalene content between 9.1 and 251.8 mg/kg (Grajzer et al., 2020). The squalene content in a solvent-extracted monovarietal oil (7.2–7.7 mg/kg oil) did not differ depending on the solvent (among five) used but was significantly lower (5.1 mg/kg oil) in cold pressed oil. (Gao et al., 2019b). Recently, five methods for the extraction of walnut oil

Table 3
Relative percentages of triacylglycerols (OLL, PLL, OOL, PLO, POO) for the studied cultivars.

Variety	Country	N	OLL				PLL				OOL				PLO				POO				Oil Ext. Mode	References
			Mean	Min	Max	Sdv	Mean	Min	Max	Sdv	Mean	Min	Max	Sdv	Mean	Min	Max	Sdv	Mean	Min	Max	Sdv		
Arco	Portugal	1	15.84				7.98				2.66				3.13				nd			Solvent	Amaral et al., 2004	
Chandler	China	1	11.19				13.45				4.50				4.58				1.07			Press	Gao et al., 2019c	
Franquette	Portugal, Tunisia	5	18.52	17.19	19.89	1.29	9.73	7.48	12.02	2.11	4.37	3.06	5.66	0.93	3.96	3.20	4.59	0.68	0.85			Solvent	Amaral et al., 2004 ; Bouabdallah et al., 2014	
Hartley	China, Portugal, Spain, Tunisia	4	13.92	6.4	17.83	6.51	13.44	9.72	17.59	3.95	4.89	3.33	6.32	1.50	4.05	3.62	4.78	0.63	0.87	0.29	1.60	0.67	Solvent, Press	Amaral et al., 2004 ; Bouabdallah et al., 2014 ; Rabadán et al., 2018b ; Gao et al., 2019c
Huguet	Spain	1									7.40								1.98			Press	Rabadán et al., 2018d	
Lara	Portugal	4	16.42	13.73	20.52	2.99	8.47	6.82	9.15	1.11	2.69	1.75	4.48	1.24	3.16	2.80	3.56	0.34	nd			Solvent	Amaral et al., 2004	
Lauzerone	Tunisia	1	21.00				12.3				7.97				5.69				1.18			Unknown	Bouabdallah et al., 2014	
Marbot	Portugal	2	19.09	16.03	23.35	3.81	8.98	7.94	9.58	0.9	3.81	2.58	6.24	2.1	3.8	3.45	4.32	0.46	nd			Solvent	Amaral et al., 2004	
Mayette	Portugal, Spain	5	17.12	14.11	22.93	4.00	9.16	6.51	11.00	1.96	4.46	1.61	8.15	3.14	3.26	2.56	3.67	0.49	2.76			Solvent, Press	Amaral et al., 2004 ; Rabadán et al., 2018d	
Mellanaise	Portugal	4	20.49	18.26	22.76	1.87	8.73	7.87	9.47	0.79	4.64	3.68	5.72	0.87	4.09	3.50	4.47	0.42	nd			Solvent	Amaral et al., 2004	
Nerpio	Spain	1	6.53				nd				nd				nd				1.12			Press	Rabadán et al., 2018d	
Parisienne	Portugal, Spain, Tunisia	6	20.51	15.86	27.57	4.45	8.83	4.83	12.07	2.73	6.63	2.99	11.49	3.52	3.92	3.19	4.60	0.55	2.48	0.89	4.07	2.25	Solvent, Press	Amaral et al., 2004 ; Bouabdallah et al., 2014 ; Rabadán et al., 2018d
Payne	Spain	1	nd				nd				6.47				nd				1.21			Press	Rabadán et al., 2018d	
Pedro	Spain	1	nd				nd				5.38				nd				1.05			Press	Rabadán et al., 2018d	
Rego	Portugal	1	14.55				7.42				2.71				2.66				nd			Solvent	Amaral et al., 2004	
Scharch-Franquette	Spain	1	nd				nd				9.84				nd				4.22			Press	Rabadán et al., 2018b	
Serr	Spain	1	nd				nd				7.94				nd				2.40			Press	Rabadán et al., 2018d	
Xiangling	China	6	10.51	7.77	12.9	1.91	15.27	13.73	16.23	0.85	2.66	1.13	4.34	1.24	5.47	4.97	5.99	0.41	1.26	0.76	1.89	0.47	Press	Gao et al., 2019a
Yunxin	China	1	12.15				11.69				3.59				5.01				1.44			Press	Gao et al., 2019c	
E cvs	China	5	10.34	9.65	11.8	0.77	10.89	5.96	14.98	3.61	2.91	2.31	3.52	0.43	4.19	2.45	5.25	1.06	1.23	1.01	1.52	0.18	Press	Gao et al., 2018
NW cvs	China	5	11.08	10.22	12.16	0.92	11.94	9.87	12.77	1.39	5.7	2.95	11.94	4.26	5.38	4.7	7.12	1.16	2.44	1.15	4.32	1.48	Press	Gao et al., 2018
SW cvs	China	5	12.52	11.15	15.46	1.71	10.95	9.38	12.09	1.01	4.62	3.54	6.98	1.37	5.09	4.67	5.41	0.34	1.60	1.21	1.92	0.31	Press	Gao et al., 2018
Unknown	Portugal, Spain	6	15.98	13.85	19.6	2.28	11.58	7.70	13.74	2.44	7.25	7.20	7.30	0.07	5.33	4.36	6.60	0.91	nd			Press	Oliveira et al., 2002 ; Bada et al., 2010	

L: linoleoyl; Ln: linolenoyl; O: oleoyl; P: palmitoyl; Mean: mean value or value when single sample; Min: minimum value; Max: maximum value; N: sample number; Sdv standard deviation; nd: not detected; Oil Ext. Mode: Oil extraction mode; E cvs: Eastern cultivars; NW cvs: Northwestern cultivars; SW cvs: Southwestern cultivars.

were compared. The highest squalene content was obtained in the roast pressing oil (14.2 mg/kg oil) and the lowest in the cold pressing oil (8.3 mg/kg oil). The differences in squalene content in oils obtained with SBE (11.9 mg/kg oil) and SC-CO₂ (6.4 mg/kg oil) showed the interest of a new green production with SBE (Gao et al., 2021). Cittadini et al. (2020) did not detect squalene in oils of six walnut genotypes, which, for them, is not surprising since this compound was barely present in nut oils according to previous works (Maguire et al., 2004). They hypothesised that the content of squalene, a precursor in the biosynthesis of sterols, could depend on the state of ripeness of the walnuts.

14. Volatile compounds

Few studies were carried out on volatile compounds in walnut oils. Different profiles were reported depending on the extraction process and the analytical method used to qualify or quantify them.

The first studies on volatile compounds were performed by Clark and Nursten, in a first time, by solvent extraction and analyses by GC/MS with a radius head on 2 columns (coated with polyethylene glycol and dimethylpolysiloxane) (Clark and Nursten, 1976). More recently, the volatile compounds were most of the time extracted by headspace-solid-phase micro-extraction (HS-SPME). The oils (0.3 g to 7.5 ml) were sealed in 10 to 22 ml headspace vials equipped with fibres coated with polydimethylsiloxane (Crowe et al., 2002; Crowe and White, 2003), polydimethylsiloxane-divinylbenzene (Bakkalbaş, 2019) or divinylbenzene-carboxene-polydimethylsiloxane (Torres et al., 2005; Martínez et al., 2006, 2010; Martínez and Maestri, 2008; Uriarte et al., 2011; Zhou et al., 2016, 2018; Ojeda-Amador et al., 2018; Grilo and Wang, 2021). The SPME fibre length ranged from 1 to 2 cm and the film thickness from 50/30 to 100 µm. The fibres were placed in the headspace at 40 or 50 °C for 5 to 60 min. The volatile compounds trapped on the fibres were desorbed directly into the GC injector for 1 to 10 min (when specified). They were analysed either by GC-FID or by GC-MS, or by both techniques, on columns, 25, 30 or 60 m long, 0.25 or 0.32 mm i. d., coated with a 0.1, 0.25 or 0.5 µm thick film consisting of polyethylene glycol, polydimethyl siloxane or 5% phenyl - methylpolysiloxane. Miraliakbari and Shahidi (2008b) analysed volatile aldehydes from 2 ml oil samples placed in 5 ml sealed headspace vials in static headspace gas chromatographic mode, with a capillary column (30 m x 0.32 mm) coated with polyethylene glycol film (0.10 µm) and an headspace sampler (Miraliakbari and Shahidi, 2008b). To avoid off-line pre-separation for the analysis of volatile compounds, an alternative approach using on-line coupled RP-HPLC and GC allowed the direct injection of the oil sample (Caja et al., 2000).

Some authors focused their studies on oil from specific walnut varieties (Torres et al., 2005; Martínez et al., 2006, 2010; Martínez and Maestri, 2008; Ojeda-Amador et al., 2018; Grilo and Wang, 2021), others compared the aroma profile of walnut oil and that of various vegetable oils (olive, almond, hazelnut, peanut and others) (Caja et al., 2000; Uriarte et al., 2011; Kesen et al., 2018). The presence of some aldehyde compounds was significantly greater in cold pressing oil than in SC-CO₂ extracted oil (Crowe and White, 2003). The volatile composition in oil differed depending on the walnut cultivars and on the environmental conditions of different crop years (Martínez et al., 2006; Ojeda-Amador et al., 2018). The volatile composition from commercial and varietal walnut oils shows wide variation in the types of compounds detected (alkanes, alcohols, aldehydes, acids, ketones, terpenes and furan derivatives) (Torres et al., 2005; Martínez et al., 2010; Ojeda-Amador et al., 2018; Zhou et al., 2018).

Depending on the oil extraction process, the oil oxidation state and geographic origin, the compounds identified differed, and so did their contents, which were expressed differently according to the studies (peak area %, mg or µg/kg oil, ppm.). Saturated and unsaturated aldehydes were the main volatile compounds. Some of them were produced from the chemical degradation of FAs (mainly linoleic acid). Others were generated through an enzymatic pathway due to the presence of

lipoxygenases, able to produce hydroperoxide precursors to cleave PUFA into short and medium chain hydrocarbons, aldehydes and alcohols (in the same manner as for the chemical auto-oxidation) (Martínez et al., 2010; Ojeda-Amador et al., 2018). (C5-C12) aldehydes were generally detected (Caja et al., 2000; Martínez and Maestri, 2008; Kesen et al., 2018; Ojeda-Amador et al., 2018) and seldom (C3-C4) aldehydes (Uriarte et al., 2011). Aromatic aldehydes (benzaldehyde and phenylacetaldehyde) were also detected according to the varieties. Few linear (C5-C9) hydrocarbons in significant quantities and cyclic, branched and aromatic hydrocarbons, in low proportions, were identified (Clark and Nursten, 1977; Uriarte et al., 2011). Unlike for olive oil, very few alcohols (mainly linear) and related compounds were found in walnut oils (Torres et al., 2005; Uriarte et al., 2011). Aromatic alcohols were also quantified (Kesen et al., 2018). Volatile acids (from formic to nonanoic acid) have been identified in the headspace of walnut oils, with a large concentration of acetic acid in many cases. Other compound families, such as pyrazine, terpenes, ketones, lactones and furans, were detected (Clark and Nursten, 1976; Caja et al., 2000; Torres et al., 2005; Martínez et al., 2006; Martínez and Maestri, 2008; Martínez et al., 2010; Uriarte et al., 2011; Zhou et al., 2016; Kesen et al., 2018; Ojeda-Amador et al., 2018). Some of them (furanones, benzaldehyde, phenylacetaldehyde, pyrazine, pyridine and pyrrole derivatives) could be associated with an oxidation process and be formed during kernel roasting prior to oil extraction (Uriarte et al., 2011). The volatile composition of cold pressed oil shown the presence of ethyl acetate, 3-carene and 1-octene-3-one whereas the volatile profile of kernels was more diverse, indicating a distinctive odour between kernels and extracted oil. The concentration of volatiles identified in both oil and kernels was similar (Grilo and Wang, 2021). When oil was extracted from kernels stocked over one to seven months under light and temperature-controlled conditions, the concentration of oil volatiles doubled rapidly (after one month depending on the variety), except for 3-carene (Grilo and Wang, 2021).

Different authors detailed the possible precursors of the main volatile compounds from enzymatic and non-enzymatic processes (Torres et al., 2005; Martínez and Maestri, 2008). Some volatile hydrocarbons and volatile aldehydes were shown positively or negatively correlated with the contents of oleic acid and linoleic acid when walnut and olive oil data were compiled together (Torres et al., 2005).

Volatile compound contents were used to indicate the degree of oxidation in vegetable oils, since many of them result from the degradation of fatty acids. Often, the contents in alkanals and alkenals (such as nonanal formed primarily from the degradation of oleic acid, hexanal from linoleic acid, *trans,trans*- and *trans,cis*-2,4-heptadienal from linolenic acid) were used to track flavour deterioration during walnut oil oxidation (Crowe and White, 2003; Miraliakbari and Shahidi, 2008b; Bakkalbaş, 2019). 2-heptenal and 1-octen-3-ol were recommended as default markers to measure walnut oil quality (Zhou et al., 2018). Some of these studies revealed that the auto-oxidative (60 °C in the dark) and photo-oxidative (35 °C in the light) stability of walnut oils differed depending on the extraction process (pressing or SC-CO₂ extraction). The off-flavour intensity of oil was modified due to the increase in the contents of some volatile compounds, particularly hexanal (Crowe and White, 2003). On the opposite, the headspace aldehyde compositions of walnut oils (extracted with different solvents) showed that walnut oils enjoyed a reasonable photo-oxidative stability under the conditions examined (fluorescent radiation for 72 h). Their volatile compositions were like those observed under auto-oxidation conditions (in a forced-air oven at 60 °C for 72 h) (Miraliakbari and Shahidi, 2008b). Nevertheless, under these auto-oxidation conditions, walnut oil behaved differently than other nut oils (almond, brazil nut, hazelnut, etc.) because hexanal was the major headspace aldehyde formed in all oxidised oils except walnut oil, which contained principally propanal (Miraliakbari and Shahidi, 2008b). A microwave pretreatment of the walnut kernels for 2 min or more have generated pyrazine compounds, which improved the typical 'roasted' flavour of hydraulic pressing oils and their oxidative stability when the kernel pretreatment was applied

Table 4
Tocopherol contents (mg/kg) in oil according to walnut variety.

Variety	Country	N	α -T				β -T				γ -T				δ -T				Oil Ext. Mode	References
			Mean	Min	Max	Sdv	Mean	Min	Max	Sdv	Mean	Min	Max	Sdv	Mean	Min	Max	Sdv		
Apollo	Poland	3	33	0	78	40	0	0	0	0	35	18	53	18	29	0	47	25	Press	Pycia et al., 2019a
Arco	Portugal	1	14				1				210				9				Solvent	Amaral et al., 2005
Asni	Morocco	20	15	9	21	3	0	0	0	0	220	162	274	30	33	19	54	10	Solvent	Kodad et al., 2016
Bilecik	Turkey	1	33				0				248				25				Soxhlet	Kafkas et al., 2017
Broadview	Poland	1		3				0				63				15			Press	Pycia et al., 2019b
Büyükoba	Turkey	2	45	43	48	3	3	2	3	0	93	91	94	2	8	7	8	0	Press, Soxhlet	Ahmed et al., 2019
Chaboksar	Iran	1		25				2				310				20			Soxhlet	Gharibzahedi et al., 2014
Chandler	Argentina, China, Italy Spain, Turkey, USA	15	14	0	115	30	0	0	1	0	246	157	527	108	28	12	63	15	Press, Solvent, Enzyme	Gao et al., 2019c;González-Gómez et al., 2019;Cittadini et al., 2020;Ozcan et al., 2020;Grilo and Wang, 2021
Dublin's Glory	New Zealand	1	29				4				355				47				Solvent	Savage et al., 1999
Elit	Serbia	2	17	16	18	1	0	0	0	0	298	275	321	33	23	15	31	11	Press	Rabrenovic et al., 2011
Esterhazy	Europe	1	26				5				343				62				Solvent	Savage et al., 1999
Fernette	Argentina	1	14				0				308				20				Press	Cittadini et al., 2020
Franquette	France, Portugal, Tunisia, USA	8	12	6	19	4	1	0	1	1	235	190	304	38	32	9	41	12	Press, Solvent, Soxhlet	Abdallah et al., 2015;Amaral et al., 2005;Cittadini et al., 2020;Lavedrine et al., 1997
Gigant	Poland	1	9				0				81				12				Press	Pycia et al., 2019b
Hanfeng	China	1		13				2				270				46			Soxhlet	Wu et al., 2020
Hartley	China, France, Portugal, Spain, Tunisia, Turkey, USA	7	37	4	126	41	0	0	1	1	286	170	554	125	30	10	67	19	Press, Solvent, Soxhlet	Amaral et al., 2005;Lavedrine et al., 1997;Abdallah et al., 2015;Kafkas et al., 2017;Ojeda-Amador et al., 2018 Gao et al., 2019c
Howard	Argentina, Turkey, USA	3	22	16	28	8	0	0	0	0	252	161	309	75	33	19	56	20	Press, Soxhlet	Kafkas et al., 2017;Cittadini et al., 2020;Grilo and Wang, 2021
Jupiter	Serbia	2	18	16	20	3	0	0	0	0	275	254	296	30	15	10	20	7	Press	Rabrenovic et al., 2011
Kaman-2	Turkey	2	49	46	52	4	3	2	3	1	101	93	108	11	8	8	8	0	Press, Soxhlet	Ahmed et al., 2019
Kaman-5	Turkey	2	46	43	50	5	3	2	3	1	92	88	97	6	9	8	9	0	Press, Soxhlet	Ahmed et al., 2019
Karaj	Iran	1	30				8				320				25				Soxhlet	Gharibzahedi et al., 2014
Koszycski	Poland	1	6				0				88				14				Press	Pycia et al., 2019b
Lake	Canada, Poland	2	6	5	6	1	0	0	0	0	155	105	205	71	13	12	15	2	Solvent, Soxhlet	Li et al., 2007,Pycia et al. 2019b
Lara	Portugal, Spain	5	16	11	24	5	1	0	2	1	276	194	517	136	21	10	56	20	Press, Solvent	Amaral et al., 2005;Ojeda-Amador et al., 2018
Lauzeronne	Tunisia	1	13				3				359				44				Soxhlet	Abdallah et al., 2015
Leopold	Poland	3	10	5	16	5	0	0	0	0	27	15	50	20	10	3	22	10	Press	Pycia et al., 2019a
Li	China	2	11	11	12	0	2	1	2	0	222	205	239	24	34	32	35	2	Soxhlet	Wu et al., 2020
Liao	China	6	18	9	21	5	4	1	5	1	237	217	261	19	43	35	52	7	Soxhlet	Wu et al., 2020
Liaoruifeng	China	1	10				1				257				55				Soxhlet	Wu et al., 2020
Local pt & gd	Tunisia	2	3	2	4	2	0	0	1	0	166	163	170	5	19	17	22	3	Soxhlet	Abdallah et al., 2015
Maras	Turkey	2	32	29	34	4	0	0	0	0	233	231	236	4	29	29	29	1	Soxhlet	Kafkas et al., 2017
Marbot	Portugal	3	11	10	13	1	1	1	1	0	238	197	262	36	12	8	14	3	Solvent	Amaral et al., 2005
Mars	Portugal	1	12				1				259				13				Press	Pycia et al., 2019b
Mayette	Portugal	4	13	11	16	2	1	1	1	0	230	201	259	28	13	11	16	2	Solvent	Amaral et al., 2005
McKinster	New Zealand	1	23				4				265				30				Solvent	Savage et al., 1999
Mellanaise	Portugal	4	10	9	11	1	1	1	1	0	205	173	230	24	11	9	15	3	Solvent	Amaral et al., 2005
Meyric	New Zealand	1	28				5				305				48				Solvent	Savage et al., 1999

(continued on next page)

Table 4 (continued)

Variety	Country	N	α -T				β -T				γ -T				δ -T				Oil Ext. Mode	References
			Mean	Min	Max	Sdv	Mean	Min	Max	Sdv	Mean	Min	Max	Sdv	Mean	Min	Max	Sdv		
Midland	Turkey	1	29				0				235				26				Soxhlet	Kafkas et al., 2017
Parisienne	Portugal, Tunisia	5	12	5	17	4	1	1	2	0	224	210	253	18	19	11	45	14	Solvent, Soxhlet	Amaral et al., 2005; Abdallah et al., 2015
Rego	Portugal	1	14				1				240				15				Solvent	Amaral et al., 2005
Resovia	Poland	3	32	29	37	4	0	0	0	0	40	27	59	16	57	10	104	47	Press	Pycia et al., 2019a
Rex	New Zealand	1	15				4				234				38				Solvent	Savage et al., 1999
Sampion	Serbia	2	17	15	19	3	0	0	0	0	339	332	345	9	17	14	20	4	Press	Rabrenovic et al., 2011
Santai	China	1	17				0				235				34				Press	Zhou et al., 2016
Rego	Portugal	1	14				1				240				15				Solvent	Amaral et al., 2005
Resovia	Poland	3	32	29	37	4	0	0	0	0	40	27	59	16	57	10	104	47	Press	Pycia et al., 2019a
Rex	New Zealand	1	15				4				234				38				Solvent	Savage et al., 1999
Sampion	Serbia	2	17	15	19	3	0	0	0	0	339	332	345	9	17	14	20	4	Press	Rabrenovic et al., 2011
Santai	China	1	17				0				235				34				Press	Zhou et al., 2016
Sebin	Turkey	1	14				0				331				24				Soxhlet	Beyhan et al., 2016
Sejnovno	Serbia	2	24	21	26	4	0	0	0	0	315	309	321	8	30	26	34	6	Press	Rabrenovic et al., 2011
Sen	Turkey	1	39				0				312				41				Soxhlet	Kafkas et al., 2017
Serr	Turkey	1	31				0				191				17				Soxhlet	Kafkas et al., 2017
Stanley	New Zealand	1	29				1				329				45				Solvent	Savage et al., 1999
Tehama	USA	1	26				5				274				37				Solvent	Savage et al., 1999
Toyserkan	Iran	1	45				10				350				30				Soxhlet	Gharibzahedi et al. 2014
Trompito	Argentina	1	6				0				307				19				Press	Cittadini et al., 2020
Tulare	Argentina	1	17				0				389				18				Press	Cittadini et al., 2020
Vina	USA	1	24				4				301				49				Solvent	Savage et al., 1999
E cvs	China	6	48	10	137	46	0	0	0	0	350	269	455	78	336	257	438	76	Press	Gao et al., 2018
NW cvs	China	5	39	15	73	24	0	0	0	0	352	170	604	157	354	161	659	184	Press	Gao et al., 2018
SW cvs	China	5	52	19	106	39	0	0	0	0	248	139	365	81	238	132	350	78	Press	Gao et al., 2018
Unknown	Austria	3	0	0	0	0	0	0	0	0	224	124	328	102	38	23	54	16	Solvent	Kornsteiner et al., 2006
Unknown	Poland	8	107	207	380		13	0	107		335	207	380		45	42	54		Press	Czaplicki et al., 2011; Grajzer et al., 2020
Unknown	Portugal	2	17	8	26	12	0	0	0	0	292	249	335	61	46	45	46	1	SC-CO ₂ , Solvent	Oliveira et al., 2002
Unknown	Spain	2	65	12	69	25	0	0	0	0	482	289	677	144	53	16	107	37	Press	Bada et al., 2010; Ojeda-Amador et al., 2018
Unknown	Ukraine	1	19				0				97				0				Solvent	Derewiaka et al., 2014
Genotype	Europe	2	22	18	25	5	5	4	6	2	308	288	327	28	39	32	45	9	Solvent	Savage et al., 1999
Genotype	New Zealand	3	20	20	21	0	6	3	8	3	253	207	285	41	36	28	42	7	Solvent	Savage et al., 1999
Genotype	Poland	6	5	2	11	4	0	0	0	0	57	11	91	28	12	7	21	5	Press	Pycia et al., 2019b
Genotype	Serbia	2	20	20	20	0	0	0	0	0	358	332	384	37	22	20	24	3	Press	Rabrenovic et al., 2011
Genotype	Turkey	33	25	1	61	18	1	0	6	2	314	155	404	69	45	3	114	31	Soxhlet	Beyhan et al., 2016; Ghafoor et al., 2020; Ozcan et al., 2020

α -T: α -Tocopherol; β -T: β -Tocopherol; γ -T: γ -Tocopherol; δ -T: δ -Tocopherol; Min: minimum value; Max: maximum value; N: sample number; Sdv: standard deviation; nd: not detected; Oil Ext. Mode : Oil extraction mode; E cvs: Eastern cultivars; NW cvs: Northwestern cultivars; SW cvs: Southwestern cultivars; SBE: subcritical butane extraction; SC-CO₂: supercritical carbon dioxide extraction.

Table 5

Total phenol content (mg std / kg oil) and ortho-diphenol content (Mmol std /kg oil) in oils according to walnut variety and standard used for total phenol quantification.

Variety	N (year)	TP			OD*		References
		Standard	λ (nm)	Content	λ (nm)	Content	
Chandler	1	Caffeic acid	765	68.40			González-Gómez et al., 2019
Franquette	1(2015), 1(2016)	Caffeic acid	765	36.20; 13.13			Rabadán et al., 2018d
Hartley	1(2015), 1(2016)	Caffeic acid	765	37.90; 13.20			Rabadán et al., 2018d
Hugget	1(2015), 1(2016)	Caffeic acid	765	31.83; 10.27			Rabadán et al., 2018d
Mayette	1(2015), 1(2016)	Caffeic acid	765	40.93; 11.43			Rabadán et al., 2018d
Nerpio	1(2015), 1(2016)	Caffeic acid	765	31.50; 18.33			Rabadán et al., 2018d
Parisienne	1(2015), 1(2016)	Caffeic acid	765	37.23; 16.47			Rabadán et al., 2018d
Payne	1(2015), 1(2016)	Caffeic acid	765	19.17; 9.53			Rabadán et al., 2018d
Pedro	1(2015), 1(2016)	Caffeic acid	765	28.40; 15.50			Rabadán et al., 2018d
Serr	1(2015), 1(2016)	Caffeic acid	765	28.13; 15.47			Rabadán et al., 2018d
Unknown	7	Caffeic acid	725	59.60 – 252.10			Grajzer et al. 2020
Unknown	1	D-Catechin	720	554.00			Czaplicki et al., 2011
Adams	1	Gallic acid	725	23.13			Slatnar et al., 2015
Chaboksar	1	Gallic acid	765	180.00	370	0.16	Gharibzahedi et al., 2014
Chandler	1	Gallic acid	765	44.78			Gao et al., 2019a
	1	Gallic acid	760	26.00			Ojeda-Amador et al., 2018
	1	Gallic acid	760	102.00			Grilo and Wang, 2021
Fernette	1	Gallic acid	725	20.55			Slatnar et al., 2015
Fernor	1	Gallic acid	725	20.16			Slatnar et al., 2015
Franquette	1	Gallic acid	725	80.08			Slatnar et al., 2015
Hartley	1	Gallic acid	765	56.38			Gao et al., 2019a
	1	Gallic acid	760	17.50			Ojeda-Amador et al., 2018
Howard	1	Gallic acid	760	143.00			Grilo and Wang, 2021
Karaj	1	Gallic acid	765	240.00	370	0.22	Gharibzahedi et al., 2014
Lara	1	Gallic acid	725	24.99			Slatnar et al., 2015
	1	Gallic acid	760	13.80			Ojeda-Amador et al., 2018
Qingxiang	1(2019)	Gallic acid	765	29.70 (CP); 35.20 (RP); 45.40 (HE); 33.60 (SBE); 28.00 (SC-CO ₂)			Gao et al., 2021
Toyserkan	1(2008)	Gallic acid	765	310.00	370	0.23	Gharibzahedi et al., 2014
	1	Gallic acid	765	220.00 (CP); 220.00 (WMC); 170.00 (MAC)	370	0.35	Gharibzahedi et al., 2013
Xiangling	1(2016), 1(2017)	Gallic acid	765	26.30 - 98.10 (different solvents)			Gao et al., 2019a
Yunxin	1	Gallic acid	765	64.61			Gao et al., 2019c
Commercial	1	Gallic acid	750	320.00	370	0.20	Arranz et al., 2008
Unknown	5	Gallic acid	725	4.10-51.80			Górnaś et al., 2014 ;Ojeda-Amador et al., 2018
	1	Gallic acid	750	468.38			Xie et al., 2018

CP: cold press extraction; HE: hexane extraction; MAC: maceration in hexane; N: sample number; OD: ortho-diphenols; RP: roast-press extraction; SBE: subcritical butane extraction; SC-CO₂: supercritical carbon dioxide extraction; TP: total phenols; WMC: water / methanol / chloroform extraction; std: standard equivalent; * Catechol, hydroxytyrosol and hydroxytyrosyl acetate solutions were used as standards.

for 4 min (Zhou et al., 2016).

Volatile compounds are known to be responsible for the complex flavour of oil. They play an important role in the consumer acceptance of some taste and smell properties of food. The first sensory analyses of walnut oils revealed a correspondence between the presence of some volatile compounds and characteristic odours through sniffing of the effluent from a GC column. None within the wide range of detected outstanding nuttiness odours was of and all associated compounds were to be considered as responsible for the final odour through collective action (Clark and Nursten, 1976). The intensity of eight attributes in relation with aromatics (nutty, oxidised and painty), basic states

(sweetness and bitterness), mouth feel (pungency and astringent) and texture (oil film), was evaluated by trained panellists' expert in olive oil tasting, for fresh walnut oils and during oil storage (120 days) (Martínez et al., 2011). The nutty sensory attribute could coincide with the presence of pentanal (Clark and Nursten, 1976). Pungent and astringent organoleptic properties detected in walnuts have been shown related to the presence of polyphenolic compounds (such as ellagitannins). However, since these compounds have been absent in walnut oils, they could not be responsible of these sensory attributes detected for oils. Thus, Martínez et al. (2011) hypothesised, by analogy with olive oil sensory analysis, that trans-2-heptenal and nonanal, found as major headspace

Table 6

Pigment contents (mg/kg) in oil according to walnut variety.

Variety	N	Carotenoids		Chlorophylls		References
		content	λ (nm)	content	λ (nm)	
Chandler	1	7.30	457	9.90	670	Ojeda-Amador et al. (2018)
Franquette	1	0.52	470	0.93	670	Martínez et al. (2013)
Hartley	1	4.20	457	3.30	670	Ojeda-Amador et al. (2018)
Lara	1	6.90	457	1.60	670	Ojeda-Amador et al. (2018)
Unknown	2	0.60-2.16	440-457	0.19-12.9	670	Nosenko et al. (2019); Górnaś et al. (2014)

N: sample number.

volatile components in many walnut oils, could be responsible for the pungent flavour of walnut oil. During storage, the intensity of nutty and sweet flavour descriptors decreased while sensory scores of rancid and bitter attributes increased (Martínez et al., 2011). Other descriptors were used as green attribute (related to hexanal), citrus flavour (associated with nonanal), fruit notes (due to the presence of 1-pentanol) (Ojeda-Amador et al., 2018). A specific study of the flavour corresponding to “roasted nut” was carried out by trained panelists, from oils pressed from walnut kernels having undergone different levels of microwave pretreatment. It showed that a correlation existed between the total amount of pyrazines in walnut oil and the “roasted” flavour scores of different oil qualities (ranging from refined oil without “roasted” flavour to oil with strongly “roasted” flavour) (Zhou et al., 2016).

15. Miscellaneous

15.1. Pigments (Carotenoid, chlorophyll and flavonoids)

Few studies reported pigment contents while chlorophylls and their degradation products are known to accelerate the lipid oxidation in the presence of light and oxygen atmosphere.

Carotenoids, chlorophylls and flavonoids were analysed in walnut oils either pressed from kernels or commercially available, dissolved in hexane or cyclohexane, by spectrophotometry (Minguez-Mosquera et al., 1991; AOCS Official Method Cc 13i-96, 2017). According to the authors, carotenoids were detected at 440 to 457 nm, chlorophylls at 670 nm and flavonoids at 404 nm (Table 6). They were quantified using either a specific absorption coefficient (Martínez et al., 2013; Ojeda-Amador et al., 2018; Nosenko et al., 2019) or a calibration curve of β -carotene (Górnaś et al., 2014), pheophytin or luteolin (Grajzer et al., 2020). Differences were observed from variety to another when cold pressing oils were stocked in amber bottles without headspace (Ojeda-Amador et al., 2018). Oils purchased in manufacturer presented a large range of pigment content, but no storage condition details were known (Nosenko et al., 2019; Grajzer et al., 2020) especially since chlorophylls can be strong photosensitizers in the oxidation process. A chlorophyll content, not exceeding 50 mg/kg, is recommended to avoid a rapid oil oxidation in the presence of light (Górnaś et al., 2014). Total flavonoid content in cold-pressed walnut oils from several unspecified varieties of *Juglans regia* kernels was low and did not exceed 13.9 mg/kg luteolin equivalent but multiple regression results showed that they have significant effects on antioxidant potential in walnut oils (Grajzer et al., 2020).

15.2. Polar lipids

Oil was extracted from crushed walnut kernels by solvent extraction (petroleum ether, then methanol or chloroform / methanol). Walnut oil lipid classes were analysed on Chromarods or by HPLC using a diol grafted stationary phase and hexane / 2-propanol as solvent system, with an evaporative light scattering detector.

Polar lipids, comprising sphingolipids (73.4%) and phospholipids (26.6%), represented 3.1% of total lipids. Their percentages in walnut oil were 2.3% and 0.8%, respectively (Tsamouris et al., 2002). HPLC analysis revealed the presence of phosphatidic acid, phosphatidylcholine and phosphatidylethanolamine, but their contents were not specified (Arranz et al., 2008). The differences between the oil extraction methods and the analytical methods used in the two articles led to incomplete results, preventing the comparison of results and the precise determination of the composition of polar lipids. Further studies would be necessary to deepen the knowledge on the polar lipid composition of walnut oils.

15.3. Vitamin K1

The phyloquinone (vitamin K1) contained in a commercial walnut

oil sample was analysed by HPLC on a silica column after purification by solid phase extraction. The phyloquinone content (15 μ g/100 g oil) was determined using an internal standard (dihydro-phyloquinone) by post-column chemical reduction of phyloquinone into its fluorescent hydroquinone derivative (Ferland and Sadowski, 1992).

15.4. Minerals

The mineral content of commercial or cold-pressed nut oils have been investigated in only two studies (Cindrić et al., 2018; Grajzer et al., 2020). Fourteen elements (As, Cd, Co, Cr, Cu, Fe, Li, Mn, Mo, Ni, Pb, Se, Sr, and Zn) were analysed by ICP-AES or ICP-MS from oils mineralised using microwave-assisted acid digestion (Cindrić et al., 2018). Cu and Fe contents were also determined using electro-thermal atomic absorption spectrometry after a mineralization in a microwave-assisted digestion oven (Grajzer et al., 2020).

The contents of the 14 elements were found below the limit of detection except Fe and Zn (31.8 and 38.6 mg/kg, respectively). Their contents were lower in oils than in kernels, except for Fe, Sr and Zn. This could be due to the contamination of the oil during the extraction process (filtering, decanting) (Cindrić et al., 2018). The median content of Cu was 0.013 mg/kg (0.008–0.017) for oils from eleven walnut varieties and 0.23 mg/kg (0.17–0.34) for Fe. Suspected of increasing to the rate of lipid autooxidation of oils, the very low contents of these transition metals were not correlated to the oxidative stability of oils (Grajzer et al., 2020).

16. Conclusions

This review provides a basis for further studies aimed at the production and quality evaluation of walnut oil, which is the main component of the kernel and has high nutritional value. Chemical data have been compiled according to walnut varieties. Fatty acids, tocopherols and sterols were the most studied compounds in order to evaluate the nutritional properties and characterize oil varieties. They are few studies regarding the quantification of phenolic compounds or the characterization of volatile compounds, both responsible of oil organoleptic properties. The comparison of the chemical composition of the varieties and the statistical analyses have been difficult for different reasons: (i) the number of samples per variety was below the statistical significance level (less than 30), (ii) geographic origin, agroclimatic parameters, ripeness stage, water content or storage conditions of the kernels were often scarcely described, although these parameters could affect the chemical composition of oils, (iii) a few studies were carried out over several harvest years, although the inter-annual variability could induce a wide dispersion in chemical compositions, (iv) different analytical methods with different detection thresholds and selectivity were used, (v) the diversity of oil extraction processes. This review could be used to complete the data of the Codex Alimentarius on walnut oil, increase the knowledge to fight falsifications and elaborate standard methods for the certification of oil quality, similar to those standards used for olive oil. To improve the quality and increase shelf-life of walnut oil (known for its poor oxidative stability), additional works would be necessary to evaluate the benefit of some agro-technological practices such as nut and kernel preservation or oil storage conditions and to promote new extraction processes. The potential of some promising walnut genotypes could be also further investigated, which would at the same time promote the walnut tree biodiversity. Although the production of walnut oil is lower compared to that of traditional oilseed oils, a better knowledge could contribute to the establishment of PDOs as there are for virgin olive oil for example. These would enhance the agricultural and technical know-how of the regional specifications, and the different of organoleptic properties of walnut oils sought after by consumers.

Funding sources

This research did not receive any specific grant from funding agencies in the public, commercial, or not-for-profit sectors.

CRedit authorship contribution statement

Catherine Rébufa: Conceptualization, Methodology, Formal analysis, Investigation, Writing. **Jacques Artaud:** Conceptualization, Methodology, Formal analysis, Investigation, Writing. **Yveline le dréau:** Conceptualization, Methodology, Formal analysis, Investigation, Writing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

References

- Abdallah, I.B., Tlili, N., Martinez-Force, E., Rubio, A.G.P., Perez-Camino, M.C., Albouchi, A., Boukhchina, S., 2015. Content of carotenoids, tocopherols, sterols, triterpenic and aliphatic alcohols, and volatile compounds in six walnuts (*Juglans regia* L.) varieties. *Food Chem.* 173, 972–978. <https://doi.org/10.1016/j.foodchem.2014.10.095>.
- Ahmed, I.A.M., Al-Juhaimi, F.Y., Özcan, M.M., Osman, M.A., Gassem, M.A., Salih, H.A.A., 2019. Effects of cold-press and soxhlet extraction systems on antioxidant activity, total phenol contents, fatty acids, and tocopherol contents of walnut kernel oils. *J. Oleo Sci.* 68, 167–173. <https://doi.org/10.5650/jos.ess18141>.
- Akhiani, S., Afshari, H., Parvaneh, T., 2017. Evaluation of some phenological and pomological characteristics of selected walnut genotypes from shahroud-iran 10. *J. Nuts* 8, 21–30. <https://doi.org/10.22034/jon.2017.530389>.
- Amaral, J.S., Alves, M.R., Seabra, R.M., Oliveira, B.P.P., 2005. Vitamin E composition of walnuts (*Juglans regia* L.): a 3-year comparative study of different cultivars. *J. Agric. Food Chem.* 53, 5467–5472. <https://doi.org/10.1021/jf050342u>.
- Amaral, J.S., Casal, S., Pereira, J.A., Seabra, R.M., Oliveira, B.P.P., 2003. Determination of sterol and fatty acid compositions, oxidative stability, and nutritional value of six walnut (*Juglans regia* L.) cultivars grown in Portugal. *J. Agric. Food Chem.* 51, 7698–7702. <https://doi.org/10.1021/jf030451d>.
- Amaral, J.S., Cunha, S.C., Alves, M.R., Pereira, J.A., Seabra, R.M., Oliveira, B.P.P., 2004. Triacylglycerol composition of walnut (*Juglans regia* L.) cultivars: characterization by HPLC-ELSD and chemometrics. *J. Agric. Food Chem.* 52, 7964–7969. <https://doi.org/10.1021/jf048918n>.
- Amin, F., Masoodi, F.A., Baba, W.N., Khan, A.A., Ganie, B.A., 2017. Effect of different ripening stages on walnut kernel quality: antioxidant activities, lipid characterization and antibacterial properties. *J. Food Sci. Technol.* 54, 3791–3801. <https://doi.org/10.1007/s13197-017-2776-4>.
- AOCS Official Method Am 2-93, 1997. In: Firestone, D.E. (Ed.), *Determination of Oil Content in Oilseeds, in Official Methods and Recommended Practices of the AOCS*, 5th ed... AOCS Press, Champaign IL. accessed 4.18.21. (<https://myaccount.aocs.org/PersonifyEbusiness/Store/ProductDetails/productId/111437>).
- AOCS Official Method Cc 131-96, 2017. Determination of chlorophyll pigments in crude vegetable oils. (<https://myaccount.aocs.org/PersonifyEbusiness/language/es-AR/Store/ProductDetails/productId/111501>) (accessed 4.9.21).
- Arranz, S., Cert, R., Pérez-Jiménez, J., Cert, A., Saura-Calixto, F., 2008. Comparison between free radical scavenging capacity and oxidative stability of nut oils. *Food Chem* 110, 985–990. <https://doi.org/10.1016/j.foodchem.2008.03.021>.
- Ayorinde, F.O., Eribo, B.E., Balan, K.V., Johnson, J.H., Wan, L.W., 1999. Determination of major triacylglycerol components of polyunsaturated specialty oils using matrix-assisted laser desorption/ionization time-of-flight mass spectrometry. *Rapid Commun. Mass Spectrom* 937–942.
- Bada, J.C., León-Camacho, M., Prieto, M., Copovi, P., Alonso, L., 2010. Characterization of walnut oils (*Juglans regia* L.) from Asturias. Spain. *J. Am. Oil Chem. Soc.* 87, 1469–1474. <https://doi.org/10.1007/s11746-010-1629-3>.
- Bakkalbaşı, E., 2019. Oxidative stability of enriched walnut oil with phenolic extracts from walnut press-cake under accelerated oxidation conditions and the effect of ultrasound treatment. *J. Food Meas. Charact.* 13, 43–50. <https://doi.org/10.1007/s11694-018-9917-y>.
- Bernard, A., Barreneche, T., Lheureux, F., Dirlwanger, E., 2018. Analysis of genetic diversity and structure in a worldwide walnut (*Juglans regia* L.) germplasm using SSR markers. *PLoS ONE* 13, e0208021. <https://doi.org/10.1371/journal.pone.0208021>.
- Beyhan, O., Gozlekci, S., Gundogdu, M., Ercisli, S., 2016. Physico-chemical and antioxidant characteristics in fruits of walnut (*Juglans regia* L.) Genotypes from Inner Anatolia. *Not. Bot. Horti. Agrobi.* 44, 586–592. <https://doi.org/10.15835/nbha44210304>.
- Beyhan, O., Özcan, A., Özcan, H., Kafkas, E., Kafkas, S., Sutyemez, M., Ercisli, S., 2017. Fat, fatty acids and tocopherol content of several walnut genotypes. *Not. Bot. Horti. Agrobi.* 45, 437–441. <https://doi.org/10.15835/nbha45210932>.
- Bingqing, H., Zhiheng, Z., Ya, C., Lihong, W., Mingshu, L., Yukui, R., 2021. Effects of different environmental conditions on composition of walnut oil. *Fresenius Environ. Bull.* 30, 3130–3133.
- Bouabdallah, I., Bouali, I., Martinez-Force, E., Albouchi, A., Perez Camino, M.C., Boukhchina, S., 2014. Composition of fatty acids, triacylglycerols and polar compounds of different walnut varieties (*Juglans regia* L.) from Tunisia. *Nat. Prod. Res.* 28, 1826–1833. <https://doi.org/10.1080/14786419.2014.950573>.
- Brühl, L., 2016. Occurrence, determination, and assessment of mineral oils in oilseeds and vegetable oils: Mineral oil hydrocarbons in oil seeds and vegetable oils. *Eur. J. Lipid Sci. Technol.* 118, 361–372. <https://doi.org/10.1002/ejlt.201500528>.
- Bujdosó, G., Kónya, É., Berki, M., Nagy-Gasztonyi, M., Bartha-Szügyi, K., Marton, B., Izsépi, F., Adányi, N., 2016. Fatty acid composition, oxidative stability, and antioxidant properties of some Hungarian and other Persian walnut cultivars. *Turk. J. Agric. For.* 160–168.
- Bujdosó, G., Tóth-Markus, M., Daood, H., Adányi, N., Szentiványi, P., 2010. Fruit quality and composition of Hungarian bred walnut cultivars. *Acta Aliment* 39, 35–47. <https://doi.org/10.1556/AAlim.39.2010.1.4>.
- Caja, M.M., Ruiz del Castillo, M.L., Martínez Alvarez, R., Herraiz, M., Blanch, G.P., 2000. Analysis of volatile compounds in edible oils using simultaneous distillation-solvent extraction and direct coupling of liquid chromatography with gas chromatography. *Eur. Food Res. Technol.* 211, 45–51. <https://doi.org/10.1007/s002170050587>.
- Caspermeyer, J., 2019. Scientists crack origin of the Persian Walnut. *Mol. Biol. Evol.* 36, 2630. <https://doi.org/10.1093/molbev/msz164>.
- Christopoulos, M.V., Tsantili, E., 2015. Oil composition in stored walnut cultivars-quality and nutritional value: Oil composition in stored walnut cultivars. *Eur. J. Lipid Sci. Technol.* 117, 338–348. <https://doi.org/10.1002/ejlt.201400082>.
- Christopoulos, M.V., Tsantili, E., 2012. Storage of fresh walnuts (*Juglans regia* L.) – Low temperature and phenolic compounds. *Postharvest Biol. Technol.* 73, 80–88. <https://doi.org/10.1016/j.postharvbio.2012.06.001>.
- Christopoulos, M.V., Tsantili, E., 2011. Effects of temperature and packaging atmosphere on total antioxidants and colour of walnut (*Juglans regia* L.) kernels during storage. *Sci. Hortic.* 131, 49–57. <https://doi.org/10.1016/j.scienta.2011.09.026>.
- Cindrić, J.I., Zeiner, M., Hlebec, D., 2018. Mineral composition of elements in walnuts and walnut oils. *IJERPH* 15, 2674. <https://doi.org/10.3390/ijerph15122674>.
- Cittadini, M.C., Martín, D., Gallo, S., Fuente, G., Bodoira, R., Martínez, M., Maestri, D., 2020. Evaluation of hazelnut and walnut oil chemical traits from conventional cultivars and native genetic resources in a non-traditional crop environment from Argentina. *Eur. Food Res. Technol.* 246, 833–843. <https://doi.org/10.1007/s00217-020-03453-8>.
- Clark, R.G., Nursten, H.E., 1977. The sensory analysis and identification of volatiles from walnut (*Juglans regia* L.) headspace. *J. Sci. Food Agric.* 28, 69–77. <https://doi.org/10.1002/jsfa.2740280111>.
- Clark, R.G., Nursten, H.E., 1976. Volatile flavour components of walnuts (*Juglans regia* L.). *J. Sci. Food Agric.* 27, 902–908. <https://doi.org/10.1002/jsfa.2740271003>.
- Copolovici, D., Bungau, S., Boscencu, R., Tit, D.M., Copolovici, L., 2017. The fatty acids composition and antioxidant activity of walnut cold press oil. *Rev. Chim.* 68, 507–509. <https://doi.org/10.37358/RC.17.3.5489>.
- Crews, C., Hough, P., Godward, J., Brereton, P., Lees, M., Guiet, S., Winkelmann, W., 2005. Study of the main constituents of some authentic walnut oils. *J. Agric. Food Chem.* 53, 4853–4860. <https://doi.org/10.1021/jf0478354>.
- Crowe, T.D., Crowe, T.W., Johnson, L.A., White, P.J., 2002. Impact of extraction method on yield of lipid oxidation products from oxidized and unoxidized walnuts. *J. Amer. Oil Chem. Soc.* 79, 453–456. <https://doi.org/10.1007/s11746-002-0505-7>.
- Crowe, T.D., White, P.J., 2003. Oxidative stability of walnut oils extracted with supercritical carbon dioxide. *J. Amer. Oil Chem. Soc.* 80, 575–578. <https://doi.org/10.1007/s11746-003-0740-y>.
- Cunniff, P., 1995. *Official Methods of Analysis of AOAC International*. Association of Official Analytical Chemists, Washington, DC.
- Czaplicki, S., Odrogowska, D., Derewiaka, D., Tańska, M., Zadernowski, R., 2011. Bioactive compounds in unsaponifiable fraction of oils from unconventional sources. *Eur. J. Lipid Sci. Technol.* 113, 1456–1464. <https://doi.org/10.1002/ejlt.201000410>.
- Derewiaka, D., Szwed, E., Wolosiak, R., 2014. Physicochemical properties and composition of lipid fraction of selected edible nuts. *Pak. J. Bot.* 46, 337–343.
- Dogan, M., Akgul, A., 2005. Fatty acid composition of some walnut (*Juglans regia* L.) cultivars from east Anatolia. *Grasas y Aceites* 56, 328–331. <https://doi.org/10.3989/gya.2005.v56.i4.101>.
- Dordoni, R., Roberta, Maggiore, Marika, Casali, Milena, Roda, Arianna, Lambri, De Faveri, Milena, dante, Marco, 2015. Effect of technological treatments and polysaccharide ingredients on oxidative stability of innovative freeze-dried walnut products. *Chem. Eng. Trans.* 43, 43–48. <https://doi.org/10.33031/CET1543008>.
- Dusek, M., Masan, V., Hic, P., Kiss, T., 2017. The evaluation of walnut oil extraction parameters. *MendelNet* 526–531.
- Erturk, U., Solak, A.O., Yucel, S., Saliyeva, K., Severoglu, Z., Ozyigit, I.I., Borkoyev, B., 2020. Determination of nut properties and chemical compositions of walnut (*Juglans regia* L.) genotypes grown in walnut-fruit forests of Kyrgyzstan. *Fresenius Environ. Bull.* 29, 2809–2817.
- Esteki, M., Farajmand, B., Amanifard, S., Barkhordari, R., Ahadiyan, Z., Dashtaki, E., Mohammadi, M., Vander Heyden, Y., 2017. Classification and authentication of Iranian walnuts according to their geographical origin based on gas chromatographic fatty acid fingerprint analysis using pattern recognition methods. *Chemom. Intell. Lab. Syst.* 171, 251–258. <https://doi.org/10.1016/j.chemolab.2017.10.014>.
- Feng, X., Zhou, H., Zulfikar, S., Luo, X., Hu, Y., Feng, L., Malvoti, M.E., Woeste, K., Zhao, P., 2018. The phylogeographic history of common walnut in China. *Front. Plant Sci.* 9, 1399. <https://doi.org/10.3389/fpls.2018.01399>.

- Ferland, G., Sadowski, J.A., 1992. Vitamin k1 (phylloquinone) content of edible oils: effect of heating and light exposure. *J. Agric. Food Chem.*, *J. Agric. Food Chem.* 40, 1869–1873.
- Gao, P., Cao, Y., Liu, R., Jin, Q., Wang, X., 2019a. Phytochemical content, minor-constituent compositions, and antioxidant capacity of screw-pressed walnut oil obtained from roasted kernels. *Eur. J. Lipid Sci. Technol.* 121, 1800292 <https://doi.org/10.1002/ejlt.201800292>.
- Gao, P., Jin, J., Liu, R., Jin, Q., Wang, X., 2018. Chemical compositions of walnut (*Juglans regia* L.) oils from different cultivated regions in China. *J. Am. Oil Chem. Soc.* 95, 825–834. <https://doi.org/10.1002/aocs.12097>.
- Gao, P., Liu, R., Jin, Q., Wang, X., 2019b. Comparison of solvents for extraction of walnut oils: Lipid yield, lipid compositions, minor-component content, and antioxidant capacity. *LWT* 110, 346–352. <https://doi.org/10.1016/j.lwt.2019.04.100>.
- Gao, P., Liu, R., Jin, Q., Wang, X., 2019c. Comparative study of chemical compositions and antioxidant capacities of oils obtained from two species of walnut: *Juglans regia* and *Juglans sigillata*. *Food Chem* 279, 279–287. <https://doi.org/10.1016/j.foodchem.2018.12.016>.
- Gao, P., Liu, R., Jin, Q., Wang, X., 2021. Effects of processing methods on the chemical composition and antioxidant capacity of walnut (*Juglans regia* L.) oil. *LWT-Food Sci. Technol.* 135, 109958. <https://doi.org/10.1016/j.lwt.2020.109958>.
- Geng, Q., Chen, J., Guo, R., Zhang, L., Li, Q., Yu, X., 2020. Salt-assisted aqueous extraction combined with Span 20 allow the obtaining of a high-quality and yield walnut oil. *LWT* 121, 108956. <https://doi.org/10.1016/j.lwt.2019.108956>.
- Ghafoor, K., Al Juhaimi, F., Gecgel, U., Babiker, E.E., Ozcan, M.M., 2020. Influence of roasting on oil content, bioactive components of different walnut kernel. *J. Oleo Sci.* 69, 423–428. <https://doi.org/10.5650/jos.ess19205>.
- Gharibzahedi, S.M.T., Mousavi, S.M., Hamed, M., Khodaiyan, F., 2014. Determination and characterization of kernel biochemical composition and functional compounds of Persian walnut oil. *J. Food Sci. Technol.* 51, 34–42. <https://doi.org/10.1007/s13197-011-0481-2>.
- Gharibzahedi, S.M.T., Mousavi, S.M., Hamed, M., Rezaei, K., Khodaiyan, F., 2013. Evaluation of physicochemical properties and antioxidant activities of Persian walnut oil obtained by several extraction methods. *Ind. Crops Prod.* 45, 133–140. <https://doi.org/10.1016/j.indcrop.2012.11.040>.
- González-Gómez, D., Ayuso-Yuste, M.C., Blanco-Roque, C., Bernalte-García, M.J., 2019. Optimization of enzyme-assisted aqueous method for the extraction of oil from walnuts using response surface methodology. *J. Food Process Preserv.* 43. <https://doi.org/10.1111/jfpp.14218>.
- Górnas, P., Siger, A., Juhnpeviča, K., Lācis, G., Šnē, E., Segliņa, D., 2014. Cold-pressed Japanese quince (*Chaenomeles japonica* (Thunb.) Lindl. ex Spach) seed oil as a rich source of α -tocopherol, carotenoids and phenolics: A comparison of the composition and antioxidant activity with nine other plant oils: The comparison of new cold-pressed Japanese quince. *Eur. J. Lipid Sci. Technol.* 116, 563–570. <https://doi.org/10.1002/ejlt.201300425>.
- Grajzer, M., Szmalczyk, K., Kuzminski, L., Witkowski, M., Kulma, A., Prescha, A., 2020. Characteristics and antioxidant potential of cold-pressed oils-possible strategies to improve oil stability. *Foods* 9, 1630. <https://doi.org/10.3390/foods9111630>.
- Greve, L.C., McGranahan, G., Hasey, J., Snyder, R., Kelly, K., Goldhamer, D., Labavitch, J.M., 1992. Variation in polyunsaturated fatty acids composition of persian walnut. *J. Amer. Soc. Hort. Sci.* 117, 518–522. <https://doi.org/10.21273/JASHS.117.3.518>.
- Grilo, F.S., Wang, S.C., 2021. Walnut (*Juglans regia* L.) volatile compounds indicate kernel and oil oxidation. *Foods* 10, 329. <https://doi.org/10.3390/foods10020329>.
- Grosso, A.L., Asensio, C.M., Grosso, N.R., Nepote, V., 2017. Sensory quality preservation of coated walnuts. *J. Food Sci* 82, 185–193. <https://doi.org/10.1111/1750-3841.13570>.
- Grosso, A.L., Asensio, C.M., Nepote, V., Grosso, N.R., 2018. Quality preservation of walnut kernels using edible coatings. *Grasas y Aceites* 69, 281. <https://doi.org/10.3989/gya.0350181>.
- Gu, X., Zhang, L., Li, L., Ma, N., Tu, K., Song, L., Pan, L., 2018. Multisource fingerprinting for region identification of walnuts in Xinjiang combined with chemometrics. *J. Food Process Eng.* 41, e12687 <https://doi.org/10.1111/jfpe.12687>.
- Han, Y., Zheng, Y., Li, S., Mo, R., Long, X., Liu, Y., 2019. Effects of drying process with different temperature on the nutritional qualities of walnut (*Juglans regia* L.). *Food Sci. Technol. Res* 25, 167–177. <https://doi.org/10.3136/fstr.25.167>.
- Hao, J., Xu, X.L., Jin, F., Regenstein, J.M., Wang, F.J., 2020. HS-SPME GC-MS characterization of volatiles in processed walnuts and their oxidative stability. *J. Food Sci. Technol.* 57, 2693–2704. <https://doi.org/10.1007/s13197-020-04305-9>.
- Helrich, K., 1990. Official methods of analysis of the AOAC. Washington, DC.
- Holčápek, M., Lísá, M., Jandera, P., Kabátová, N., 2005. Quantitation of triacylglycerols in plant oils using HPLC with APCL-MS, evaporative light-scattering, and UV detection. *J. Sep. Sci.* 28, 1315–1333. <https://doi.org/10.1002/jssc.200500088>.
- Horwitz, W., Latimer, G.W., 2005. Official methods of analysis of AOAC International. AOAC International, Gaithersburg, Md.
- Iqbal, M., Bhatti, I.A., Shahid, M., Nisar, J., 2016. Physicochemical characterization, microbial decontamination and shelf life analysis of walnut (*Juglans regia* L.) oil extracted from gamma radiation treated seeds. *Biocatal. Agric. Biotechnol.* 6, 116–122. <https://doi.org/10.1016/j.cbab.2016.03.004>.
- ISO 659, 1998, ISO 659 - Graines oléagineuses - Détermination de la teneur en huile (Méthode de référence). (<https://www.iso.org/cms/render/live/fr/sites/isoorg/contents/data/standard/02/32/23297.html>) (accessed 3.20.21).
- ISO 734-1, 1998, Oilseed meals-determination of oil content. iTech Standards Store. URL (<https://standards.iteh.ai/catalog/standards/iso/e2239b57-bf72-491f-a3a7-c5c00cbd5397/iso-734-1-1998>) (accessed 7.8.21).
- ISO 5509:2000, 2000, ISO 5509:2000 - Corps gras d'origines animale et végétale -Préparation des esters méthyliques d'acides gras. (<https://www.iso.org/cms/render/live/fr/sites/isoorg/contents/data/standard/01/15/11560.html>) (accessed 3.8.21).
- IUPAC Method 2.210, 1992. IUPAC. (International Union of Pure and Applied Chemistry). Standard methods for the analysis of oils, fats and derivatives: Method 2.210. Determination of Fatty Acids in The 2-Position of the Triglycerides of Oils and Fats. 1st Suppl, 7th ed., Pergamon Press., Oxford, U.K.
- IUPAC No. 2301, 1987. IUPAC (International Union of Pure and Applied Chemistry) No. 2301. Preparation of the fatty acid methyl esters. Standard methods for the analysis of oils, fats and derivatives. International Union of Pure and Applied Chemistry, Blackwell., Oxford, UK.
- Jahanban-Esfahlan, A., Ostadrahimi, A., Tabibiazar, M., Amarowicz, R., 2019. A comparative review on the extraction, antioxidant content and antioxidant potential of different parts of walnut (*Juglans regia* L.) fruit and tree. *Molecules* 24, 2133. <https://doi.org/10.3390/molecules24112133>.
- Jaiswal, B.S., Tailang, M., 2017. *Juglans regia*: a review of its traditional uses. *Indo Am. J. Pharm. Res.* 7, 9.
- Jie, Y., Zhao, H., Zhang, B., 2019. The role of an acidic peptide in controlling the oxidation process of walnut oil. *Foods* 8, 499. <https://doi.org/10.3390/foods8100499>.
- JORF n°0271, 2018, Arrêté du 16 novembre 2018 relatif à l'appellation d'origine contrôlée " Huile de noix du Périgord " (No. Texte n°30-NOR: AGRT1829077A).
- Kafkas, E., Attar, S.H., Gundesli, M.A., Ozcan, A., Ergun, M., 2020. Phenolic and fatty acid profile, and protein content of different walnut cultivars and genotypes (*Juglans regia* L.) grown in the USA. *Int. J. Fruit Sci* 20, S1711–S1720. <https://doi.org/10.1080/15538362.2020.1830014>.
- Kafkas, E., Burgut, A., Ozcan, H., Ozcan, A., Sutyemez, M., Kafkas, S., Türemis, N., 2017. Fatty acid, total phenol and tocopherol profiles of some walnut cultivars: a comparative study. *FNS* 08, 1074–1084. <https://doi.org/10.4236/fns.2017.812079>.
- Kesen, S., Amanpour, A., Selli, S., 2018. Comparative evaluation of the fatty acids and aroma compounds in selected Iranian nut oils. *Eur. J. Lipid Sci. Technol.* 120, 1800152 <https://doi.org/10.1002/ejlt.201800152>.
- Kodad, O., Estopañán, G., Juan, T., Socias i Company, R., Sindic, M., 2016. Genotype and year variability of the chemical composition of walnut oil of Moroccan seedlings from the high Atlas Mountains. *Grasas y Aceites* 67, e116. <https://doi.org/10.3989/gya.0256151>.
- Kornsteiner, M., Wagner, K.-H., Elmadafa, I., 2006. Tocopherols and total phenolics in 10 different nut types. *Food Chem* 98, 381–387. <https://doi.org/10.1016/j.foodchem.2005.07.033>.
- Koyuncu, M.A., Yarılgac, T., Kazankaya, A., Koyuncu, F., Küçük, M., Sen, S.M., 2001. Compositional changes of fatty acids during the development of kernel of Yalova-1 and Yalova-4 walnut cultivars. *Acta Hort* 585–589. <https://doi.org/10.17660/ActaHortic.2001.544.81>.
- Laskos, K., Pisulewska, E., Waligorski, P., Janowiak, F., Janeczko, A., Sadura, I., Polaszczyk, S., Czyczyło-Mysza, I.M., 2021. Herbal additives substantially modify antioxidant properties and tocopherol content of cold-pressed oils. *Antioxidants* 10, 781. <https://doi.org/10.3390/antiox10050781>.
- Lavedrine, F., Ravel, A., Poupard, A., Alary, J., 1997. Effect of geographic origin, variety and storage on tocopherol concentrations in walnuts by HPLC. *Food Chem* 58, 135–140. [https://doi.org/10.1016/S0308-8146\(96\)00232-4](https://doi.org/10.1016/S0308-8146(96)00232-4).
- Leahu, A., Damian, C., Oroian, M., Hreţcanu, E., 2013. Estimation of biochemical properties of walnuts from the region of Suceava - Romania. *Food and Environment Safety - Journal of Faculty of Food Engineering, Ştefan cel Mare University - Suceava, Food and Environment Safety - J. Food Eng* 169–175.
- Leahu, A., Oroian, M., Ropciuc, S., 2016. The quality and stability of walnut oil under the influence of storage conditions. *Scientific Papers. Animal Science Series: Lucrări Ştiinţifice - Seria Zootehnie. USAMV Iasi* 65, 230–236.
- Li, L., Tsao, R., Yang, R., Kramer, J.K.G., Hernandez, M., 2007. Fatty acid profiles, tocopherol contents, and antioxidant activities of heartnut (*Juglans ailanthifolia* Var. cordiformis) and Persian walnut (*Juglans regia* L.). *J. Agric. Food Chem* 55, 1164–1169. <https://doi.org/10.1021/jf062322d>.
- Li, Q., Yin, R., Zhang, Q., Wang, X., Hu, X., Gao, Z., Duan, Z., 2017. Chemometrics analysis on the content of fatty acid compositions in different walnut (*Juglans regia* L.) varieties. *Eur. Food Res. Technol.* 243, 2235–2242. <https://doi.org/10.1007/s00217-017-2925-z>.
- Liang, P., Chen, C., Zhao, S., Ge, F., Liu, D., Liu, B., Fan, Q., Han, B., Xiong, X., 2013. Application of fourier transform infrared spectroscopy for the oxidation and peroxide value evaluation in virgin walnut oil. *J. Spectrosc.* 2013, 1–5. <https://doi.org/10.1155/2013/138728>.
- Liu, B., Liang, J., Zhao, D., Wang, K., Jia, M., Wang, J., 2020. Morphological and compositional analysis of two walnut (*Juglans regia* L.) cultivars growing in China. *Plant Foods Hum. Nutr* 75, 116–123. <https://doi.org/10.1007/s11130-019-00794-y>.
- Maestri, D., Cittadini, M.C., Bodoira, R., Martínez, M., 2020. Tree nut oils: chemical profiles, extraction, stability, and quality concerns. *Eur. J. Lipid Sci. Technol.* 122, 1900450 <https://doi.org/10.1002/ejlt.201900450>.
- Maguire, L.S., O'Sullivan, S.M., Galvin, K., O'Connor, T.P., O'Brien, N.M., 2004. Fatty acid profile, tocopherol, squalene and phytoosterol content of walnuts, almonds, peanuts, hazelnuts and the macadamia nut. *Int. J. Food Sci. Nutr.* 55, 171–178. <https://doi.org/10.1080/09637480410001725175>.
- Martínez, M.L., Bordón, M.G., Bodoira, R.M., Penci, M.C., Ribotta, P.D., Maestri, D.M., 2017. Walnut and almond oil screw-press extraction at industrial scale: effects of process parameters on oil yield and quality. *Grasas y Aceites* 68, 216. <https://doi.org/10.3989/gya.0554171>.
- Martínez, M.L., Penci, M.C., Ixtaina, V., Ribotta, P.D., Maestri, D., 2013. Effect of natural and synthetic antioxidants on the oxidative stability of walnut oil under

- different storage conditions. *LWT - Food Sci. Tech.* 51, 44–50. <https://doi.org/10.1016/j.lwt.2012.10.021>.
- Martínez, M., Barrionuevo, G., Nepote, V., Grosso, N., Maestri, D., 2011. Sensory characterisation and oxidative stability of walnut oil: sensory characterisation and oxidative stability of WO. *Int. J. Food Sci. Tech.* 46, 1276–1281. <https://doi.org/10.1111/j.1365-2621.2011.02618.x>.
- Martínez, M.L., Labuckas, D.O., Lamarque, A.L., Maestri, D.M., 2010. Walnut (*Juglans regia* L.): genetic resources, chemistry, by-products (n/a-n/a). *J. Sci. Food Agric.* <https://doi.org/10.1002/jsfa.4059>.
- Martínez, M.L., Maestri, D.M., 2008. Oil chemical variation in walnut (*Juglans regia* L.) genotypes grown in Argentina. *Eur. J. Lipid Sci. Technol.* 110, 1183–1189. <https://doi.org/10.1002/ejlt.200800121>.
- Martínez, M.L., Mattea, M.A., Maestri, D.M., 2008. Pressing and supercritical carbon dioxide extraction of walnut oil. *J. Food Eng.* 88, 399–404. <https://doi.org/10.1016/j.jfoodeng.2008.02.026>.
- Martínez, M.L., Mattea, M.A., Maestri, D.M., 2006. Varietal and crop year effects on lipid composition of walnut (*Juglans regia*) genotypes. *J. Amer. Oil Chem. Soc.* 83, 791–796. <https://doi.org/10.1007/s11746-006-5016-z>.
- Matthäus, B.W., 1996. Determination of the oxidative stability of vegetable oils by rancimat and conductivity and chemiluminescence measurements. *J. Am. Oil Chem. Soc.* 73, 1039–1043. <https://doi.org/10.1007/BF02523413>.
- Matthäus, B., Özcan, M.M., Juhaimi, F.A., Adiamo, O.Q., Alsawmahi, O.N., Ghafoor, K., Babiker, E.E., 2018. Effect of the harvest time on oil yield, fatty acid, tocopherol and sterol contents of developing almond and walnut kernels. *J. Oleo Sci.* 67, 39–45. <https://doi.org/10.5650/jos.ess17162>.
- McGill, A.S., Moffat, C.F., Mackie, P.R., Cruickshank, P., 1993. The composition and concentration of n-alkanes in retail samples of edible oils. *J. Sci. Food Agric.* 61, 357–362. <https://doi.org/10.1002/jsfa.2740610312>.
- Mexis, S.F., Badeka, A.V., Riganakos, K.A., Karakostas, K.X., Kontominas, M.G., 2009. Effect of packaging and storage conditions on quality of shelled walnuts. *Food Control* 20, 743–751. <https://doi.org/10.1016/j.foodcont.2008.09.022>.
- Minguez-Mosquera, I.M., Rejano-Navarro, L., Gandul-Rojas, B., Sanchez-Gomez, A.H., Garrido-Fernandez, J., 1991. Color-pigment correlation in virgin olive oil. *J. Am. Oil Chem. Soc.* 68, 332–336. <https://doi.org/10.1007/BF02657688>.
- Miraliakbari, H., Shahidi, F., 2008a. Lipid class compositions, tocopherols and sterols of tree nut oils extracted with different solvents. *J. Food Lipids* 15, 81–96. <https://doi.org/10.1111/j.1745-4522.2007.00104.x>.
- Miraliakbari, H., Shahidi, F., 2008b. Oxidative stability of tree nut oils. *J. Agric. Food Chem.* 56, 4751–4759. <https://doi.org/10.1021/jf8000982>.
- Moreda, V., Pérez-Camino, M.C., Cert, A., 2001. Gas and liquid chromatography of hydrocarbons in edible vegetable oils. *J. Chromatogr. A* 936, 159–171. [https://doi.org/10.1016/S0021-9673\(01\)01222-5](https://doi.org/10.1016/S0021-9673(01)01222-5).
- Nguyen, T.H.D., Vu, D.C., 2021. A review on phytochemical composition and potential health-promoting properties of walnuts. *Food Rev. Int.* <https://doi.org/10.1080/87559129.2021.1912084>.
- Nosenko, T., Koroluk, T., Usatuk, S., Vovk, G., Kostinova, T., 2019. Comparative study of the biological value and oxidative stability of walnut and pumpkin-seed oils. *Food Sci. Tech.* 13, 60–65. <https://doi.org/10.15673/fst.v13i1.1311>.
- OFAG, 2020. Appellations d'origine et indications géographiques. Office fédéral de l'agriculture. (<https://www.blw.admin.ch/blw/fr/home/instrumente/kennzeichnung/ursprungsbezeichnungen-und-geografische-angaben.html>) (accessed 3.19.21).
- Official Journal of The European Union, 2020. Huile de Noix du Périgord (No. C 308/22).
- Ojeda-Amador, R.M., Salvador, M.D., Gómez-Alonso, S., Fregapane, G., 2018. Characterization of virgin walnut oils and their residual cakes produced from different varieties. *Food Res. Int.* 108, 396–404. <https://doi.org/10.1016/j.foodres.2018.03.066>.
- Oliveira, R., Rodrigues, M.F., Bernardo-Gil, M.G., 2002. Characterization and supercritical carbon dioxide extraction of walnut oil. *J. Amer. Oil Chem. Soc.* 79, 225–230. <https://doi.org/10.1007/s11746-002-0465-y>.
- Ortiz, C.M., Vicente, A.R., Fields, R.P., Grilo, F., Labavitch, J.M., Donis-Gonzalez, I., Crisosto, C.H., 2019. Walnut (*Juglans regia* L.) kernel postharvest deterioration as affected by pellicle integrity, cultivar and oxygen concentration. *Postharvest Biol. Tec.* 156, 110948. <https://doi.org/10.1016/j.postharvbio.2019.110948>.
- Özcan, A., Sutaymez, M., Attar, S.H., Kafkas, E., Ergun, M., 2020. Fatty acid composition, phenolic compound content and antioxidant activity of unique walnut genotypes with red seed coat. *J. Food Nutr. Res.* 59, 352–360.
- Özcan, M.M., Arslan, D., 2011. Antioxidant effect of essential oils of rosemary, clove and cinnamon on hazelnut and poppy oils. *Food Chem* 129, 171–174. <https://doi.org/10.1016/j.foodchem.2011.01.055>.
- Özcan, M.M., İman, C., Arslan, D., 2010. Physicochemical properties, fatty acid and mineral content of some walnuts (*Juglans regia* L.) types. *Agr. Sci* 01, 62–67. <https://doi.org/10.4236/as.2010.12009>.
- Ozkan, G., Koyuncu, M.A., 2005. Physical and chemical composition of some walnut (*Juglans regia* L.) genotypes grown in Turkey. *Grasas y Aceites* 56, 141–146. <https://doi.org/10.3989/gya.2005.v56.i2.122>.
- Peng, J., Liu, F., Kong, W., Zhang, C., Yu, K., He, Y., 2017. Rapid identification of varieties of walnut powder based on laser-induced breakdown spectroscopy. *Trans. ASABE* 60, 19–28. <https://doi.org/10.13031/trans.11553>.
- Pereira, J.A., Oliveira, I., Sousa, A., Ferreira, I.C.F.R., Bento, A., Estevinho, L., 2008. Bioactive properties and chemical composition of six walnut (*Juglans regia* L.) cultivars. *Food Chemical Toxicol* 46, 2103–2111. <https://doi.org/10.1016/j.fct.2008.02.002>.
- Persic, M., Mikulic-Petkovsek, M., Halbwirth, H., Solar, A., Veberic, R., Slatnar, A., 2018. Red walnut: characterization of the phenolic profiles, activities and gene expression of selected enzymes related to the phenylpropanoid pathway in pellicle during walnut development. *J. Agric. Food Chem.* 66, 2742–2748. <https://doi.org/10.1021/acs.jafc.7b05603>.
- Poggetti, L., Ferfua, C., Chiabà, C., Testolin, R., Baldini, M., 2018. Kernel oil content and oil composition in walnut (*Juglans regia* L.) accessions from north-eastern Italy: kernel oil content and oil composition in Italian walnuts. *J. Sci. Food Agric.* 98, 955–962. <https://doi.org/10.1002/jsfa.8542>.
- Pycia, K., Kapusta, I., Jaworska, G., 2019a. Impact of the degree of maturity of walnuts (*Juglans regia* L.) and their variety on the antioxidant potential and the content of tocopherols and polyphenols. *Molecules* 24, 2936. <https://doi.org/10.3390/molecules24162936>.
- Pycia, K., Kapusta, I., Jaworska, G., Jankowska, A., 2019b. Antioxidant properties, profile of polyphenolic compounds and tocopherol content in various walnut (*Juglans regia* L.) varieties. *Eur Food Res Technol* 245, 607–616. <https://doi.org/10.1007/s00217-018-3184-3>.
- Rabadán, A., Álvarez-Ortí, M., Pardo, J.E., Alvarruiz, A., 2018a. Storage stability and composition changes of three cold-pressed nut oils under refrigeration and room temperature conditions. *Food Chem* 259, 31–35. <https://doi.org/10.1016/j.foodchem.2018.03.098>.
- Rabadán, A., Pardo, J.E., Gómez, R., Álvarez-Ortí, M., 2018b. Evaluation of physical parameters of walnut and walnut products obtained by cold pressing. *LWT* 91, 308–314. <https://doi.org/10.1016/j.lwt.2018.01.061>.
- Rabadán, A., Pardo, J.E., Gómez, R., Álvarez-Ortí, M., 2018c. Influence of temperature in the extraction of nut oils by means of screw pressing. *LWT* 93, 354–361. <https://doi.org/10.1016/j.lwt.2018.03.061>.
- Rabadán, A., Pardo, J.E., Pardo-Giménez, A., Álvarez-Ortí, M., 2018d. Effect of genotype and crop year on the nutritional value of walnut virgin oil and defatted flour. *Sci. Total Environ.* 634, 1092–1099. <https://doi.org/10.1016/j.scitotenv.2018.04.090>.
- Rabrenovic, B., Dimic, E., Maksimovic, M., Sobajic, S., Gajic-Krstajic, L., 2011. Determination of fatty acid and tocopherol compositions and the oxidative stability of walnut (*Juglans regia* L.) cultivars grown in Serbia. *Czech J. Food Sci* 29, 74–78. <https://doi.org/10.17221/180/2010-CJFS>.
- Rueda, A., Samaniego-Sánchez, C., Olalla, M., Giménez, R., Cabrera-Vique, C., Seiquer, I., Lara, L., 2016. Combination of analytical and chemometric methods as a useful tool for the characterization of extra virgin argan oil and other edible virgin oils. role of polyphenols and tocopherols. *J. AOAC Int.* 99, 489–494. <https://doi.org/10.5740/jaoacint.15-0121>.
- Salgun, S., Salgun, U., 2006. Supercritical fluid extraction of walnut kernel oil. *Eur. J. Lipid Sci. Technol.* 108, 577–582. <https://doi.org/10.1002/ejlt.200600046>.
- Sánchez-Morgado, J.R., García-Parra, J., Ramírez, R., Hernández, T., González-Gómez, D., 2013. Comparative study of the nutritional and bioactive compounds content of four walnut (*Juglans regia* L.) cultivars. *J. Food Compos. Anal.* 31, 232–237. <https://doi.org/10.1016/j.jfca.2013.06.004>.
- Savage, G.P., 2001. Chemical composition of walnuts (*Juglans regia* L.) grown in New Zealand. *Plant Food Hum. Nutr* 56, 75–82. <https://doi.org/10.1023/A:1008175606698>.
- Savage, G.P., Dutta, P.C., McNeil, D.L., 1999. Fatty acid and tocopherol contents and oxidative stability of walnut oils. *J. Am. Oil Chem. Soc.* 76, 1059–1063. <https://doi.org/10.1007/s11746-999-0204-2>.
- Schwartz, H., Ollilainen, V., Piironen, V., Lampi, A.-M., 2008. Tocopherol, tocotrienol and plant sterol contents of vegetable oils and industrial fats. *J. Food Compos. Anal.* 21, 152–161. <https://doi.org/10.1016/j.jfca.2007.07.012>.
- Sena-Moreno, E., Pardo, J.E., Pardo-Giménez, A., Gómez, R., Alvarez-Ortí, M., 2016. Differences in oils from nuts extracted by means of two pressure systems. *Int. J. Food Prop.* 19, 2750–2760. <https://doi.org/10.1080/10942912.2016.1144068>.
- Slatnar, A., Mikulic-Petkovsek, M., Stampar, F., Veberic, R., Solar, A., 2015. Identification and quantification of phenolic compounds in kernels, oil and bagasse pellets of common walnut (*Juglans regia* L.). *Food Res. Int.* 67, 255–263. <https://doi.org/10.1016/j.foodres.2014.11.016>.
- Somogyi, L., Gorka, Á., Soós, A., Badak-Kerti, K., Kóczán-Manninger, K., Szedljak, I., 2015. Effect of immersed herbs on the oxidative stability of edible oils. *Period. Polytech. Chem. Eng.* 59, 272–276. <https://doi.org/10.3311/PPCh.7571>.
- Tapia, M.I., Sánchez-Morgado, J.R., García-Parra, J., Ramírez, R., Hernández, T., González-Gómez, D., 2013. Comparative study of the nutritional and bioactive compounds content of four walnut (*Juglans regia* L.) cultivars. *J. Food Compos. Anal.* 31, 232–237. <https://doi.org/10.1016/j.jfca.2013.06.004>.
- Teixeira, A.M., Sousa, C., 2019. A review on the application of vibrational spectroscopy to the chemistry of nuts. *Food Chem* 277, 713–724. <https://doi.org/10.1016/j.foodchem.2018.11.030>.
- Torres, M.M., Martínez, M.L., Maestri, D.M., 2005. A multivariate study of the relationship between fatty acids and volatile flavor components in olive and walnut oils. *J. Amer. Oil Chem. Soc.* 82, 105–110. <https://doi.org/10.1007/s11746-005-1050-0>.
- Tsamouris, G., Hatziantoniou, S., Demetzos, C., 2002. Lipid Analysis of Greek Walnut Oil (*Juglans regia* L.). *Z. Naturforsch. C* 57, 51–56. <https://doi.org/10.1515/znc-2002-1-209>.
- Uriarte, P.S., Goicoechea, E., Guillen, M.D., 2011. Volatile components of several virgin and refined oils differing in their botanical origin: Volatile components of virgin and refined oils. *J. Sci. Food Agric.* 91, 1871–1884. <https://doi.org/10.1002/jsfa.4400>.
- Verardo, V., Bendini, A., Cerretani, L., Malaguti, D., Cozzolino, E., Caboni, M.F., 2009. Capillary gas chromatography analysis of lipid composition and evaluation of phenolic compounds by micellar electrokinetic chromatography in Italian walnut (*Juglans regia* L.): irrigation and fertilization influence. *J. Food Quality* 32, 262–281. <https://doi.org/10.1111/j.1745-4557.2009.00249.x>.
- Viera-Alcaide, I., Hamdi, A., Jiménez-Araujo, A., Rodríguez-Arcos, R., Espejo-Calvo, J.A., Guillén-Bejarano, R., 2019. Nutritional composition and antioxidant activity of

- different walnut varieties (*Juglans regia* L.) from Nerpio (Spain) in comparison to commercial varieties. *Grasas y Aceites* 70, 310. <https://doi.org/10.3989/gya.0932182>.
- Wang, W.Q., Yang, C.Y., Li, D.P., Wang, H.Q., Rong, R.F., 2014. Effects of different heat treatments on lipoxygenase activity and the oxidative stability of walnut oil. *Acta Horti* 249–255. <https://doi.org/10.17660/ActaHortic.2014.1050.33>.
- Wei, F., Chen, Q., Du, Y., Han, C., Fu, M., Jiang, H., Chen, X., 2020. Effects of hulling methods on the odor, taste, nutritional compounds, and antioxidant activity of walnut fruit. *LWT* 120, 108938. <https://doi.org/10.1016/j.lwt.2019.108938>.
- Wu, S., Ni, Z., Wang, R., Zhao, B., Han, Y., Zheng, Y., Liu, F., Gong, Y., Tang, F., Liu, Y., 2020. The effects of cultivar and climate zone on phytochemical components of walnut (*Juglans regia* L.). *Food Energy Secur* 00 (e196), 1–13. <https://doi.org/10.1002/fes3.196>.
- Xie, C., Ma, Z.F., Li, F., Zhang, H., Kong, L., Yang, Z., Xie, W., 2018. Storage quality of walnut oil containing lycopene during accelerated oxidation. *J. Food Sci. Technol.* 55, 1387–1395. <https://doi.org/10.1007/s13197-018-3053-x>.
- Yang, R., Zhang, L., Li, P., Yu, L., Mao, J., Wang, X., Zhang, Q., 2018. A review of chemical composition and nutritional properties of minor vegetable oils in China. *Trends Food Sci. Tech.* 74, 26–32. <https://doi.org/10.1016/j.tifs.2018.01.013>.
- Yildiz, A.Y., Karaca, H., 2021. Comparison of the oil quality of light and dark walnuts under different storage conditions. *J. Oleo Sci.* 70, 615–632. <https://doi.org/10.5650/jos.ess20266>.
- Yuan, X., Huang, S., Ma, H., Huang, N., Ye, N., 2019. Differential responses of walnut cultivars to cold storage and their correlation with postharvest physiological parameters. *Hortic. Environ. Biotechnol.* 60, 345–356. <https://doi.org/10.1007/s13580-019-00126-8>.
- Zhai, M.Z., Wang, D., Tao, X.D., Wang, Z.Y., 2015. Fatty acid compositions and tocopherol concentrations in the oils of 11 varieties of walnut (*Juglans regia* L.) grown at Xinjiang, China. *J. Horti. Sci. Biotech.* 90, 715–718. <https://doi.org/10.1080/14620316.2015.11668736>.
- Zhang, L., Wu, S., Jin, X., 2021. Fatty acid stable carbon isotope ratios combined with oxidation kinetics for characterization and authentication of walnut oils. *J. Agric. Food Chem.* 69, 6701–6709. <https://doi.org/10.1021/acs.jafc.1c01843>.
- Zhang, Y., Li, X., Lu, X., Sun, H., Wang, F., 2021. Effect of oilseed roasting on the quality, flavor and safety of oil: A comprehensive review. *Food Res. Int.* 150, 110791. <https://doi.org/10.1016/j.foodres.2021.110791>.
- Zhou, Y., Fan, W., Chu, F., Pei, D., 2016. Improvement of the flavor and oxidative stability of walnut oil by microwave pretreatment. *J. Am. Oil Chem Soc.* 93, 1563–1572. <https://doi.org/10.1007/s11746-016-2891-9>.
- Zhou, Y., Fan, W., Chu, F., Wang, C., Dong, Pei, 2018. Identification of volatile oxidation compounds as potential markers of walnut oil quality. *J. Food Sci.* 83, 2745–2752. <https://doi.org/10.1111/1750-3841.14342>.
- Zwarts, L., Savage, G.P., McNeil, D.L., 1999. Fatty acid content of New Zealand-grown walnuts (*Juglans regia* L.). *Int. J. Food Sci. Nutr.* 50, 189–194. <https://doi.org/10.1080/096374899101229>.