

Article

Variations in Antioxidant Capacity, Oxidative Stability, and Physicochemical Quality Parameters of Walnut (*Juglans regia*) Oil with Roasting and Accelerated Storage Conditions

Youssef Elouafy ¹, Zineb Lakhlifi El Idrissi ¹, Adil El Yadini ¹, Hicham Harhar ¹, Mohammed Merae Alshahrani ², Ahmed Abdullah AL Awadh ², Khang Wen Goh ^{3,*}, Long Chiau Ming ⁴, Abdelhakim Bouyahya ^{5,*} and Mohamed Tabyaoui ¹

¹ Laboratory of Materials, Nanotechnology and Environment LMNE, Faculty of Sciences, Mohammed V University in Rabat, Rabat BP 1014, Morocco

² Department of Clinical Laboratory Sciences, Faculty of Applied Medical Sciences, Najran University, P.O. Box 1988, Najran 61441, Saudi Arabia

³ Faculty of Data Science and Information Technology, INTI International University, Nilai 71800, Malaysia

⁴ PAP Rashidah Sa'adatul Bolkliah Institute of Health Sciences, Universiti Brunei Darussalam, Gadong BE1410, Brunei

⁵ Laboratory of Human Pathologies Biology, Faculty of Sciences, Mohammed V University in Rabat, Rabat BP 1014, Morocco

* Correspondence: khangwen.goh@newinti.edu.my (K.W.G.); a.bouyahya@um5r.ac.ma (A.B.)



Citation: Elouafy, Y.; El Idrissi, Z.L.; El Yadini, A.; Harhar, H.; Alshahrani, M.M.; AL Awadh, A.A.; Goh, K.W.; Ming, L.C.; Bouyahya, A.; Tabyaoui, M. Variations in Antioxidant Capacity, Oxidative Stability, and Physicochemical Quality Parameters of Walnut (*Juglans regia*) Oil with Roasting and Accelerated Storage Conditions. *Molecules* **2022**, *27*, 7693. <https://doi.org/10.3390/molecules27227693>

Academic Editors:
Domenico Montesano,
Riccardo Petrelli and
Nazimah Hamid

Received: 29 August 2022

Accepted: 16 September 2022

Published: 9 November 2022

Publisher's Note: MDPI stays neutral with regard to jurisdictional claims in published maps and institutional affiliations.



Copyright: © 2022 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

Abstract: Walnut oil, like all vegetable oils, is chemically unstable because of the sensitivity of its unsaturated fatty acids to the oxidation phenomenon. This phenomenon is based on a succession of chemical reactions, under the influence of temperature or storage conditions, that always lead to a considerable change in the quality of the oil by promoting the oxidation of unsaturated fatty acids through the degradation of their C–C double bonds, leading to the formation of secondary oxidation products that reduce the nutritional values of the oil. This research examines the oxidative stability of roasted and unroasted cold-pressed walnut oils under accelerated storage conditions. The oxidative stability of both oils was evaluated using physicochemical parameters: chemical composition (fatty acids, phytosterols, and tocopherols), pigment content (chlorophyll and carotenoids), specific extinction coefficients (K_{232} and K_{270}), and quality indicators (acid and peroxide value) as well as the evaluation of radical scavenging activity by the DPPH method. The changes in these parameters were evaluated within 60 days at 60 ± 2 °C. The results showed that the levels of total phytosterols, the parameters of the acid and peroxide value, K_{232} and K_{270} , increased slightly for both oils as well as the total tocopherol content and the antioxidant activity affected by the roasting process. In contrast, the fatty acid profiles did not change considerably during the 60 days of our study. After two months of oil treatment at 60 °C, the studied oils still showed an excellent physicochemical profile, which allows us to conclude that these oils are stable and can withstand such conditions. This may be due to the considerable content of tocopherols (vitamin E), which acts as an antioxidant.

Keywords: *Juglans regia*; walnut oil; oxidative stability; roasting; antioxidant activity; DPPH; tocopherol; phytosterol

1. Introduction

The common walnut (*Juglans regia* L.), Juglandaceae family, known also as English or Persian walnut, is a source of many bioactive compounds in many plant parts including the nut (seeds or kernels), leaves, flowers, shell, root, or bark, used in food, pharmaceutical, and cosmetic industries [1].

Juglans regia walnut oil, alongside its important economic value [2], is considered one of the richest oils in vitamins and antioxidant compounds [3], giving it many advantages in resistance to oxidation [4]. Walnut seeds represent a solid source of nutritional

compounds [5] with several biological properties including antioxidant activity [6–12], anti-inflammatory activity [13–20], antibacterial activity [21–23], neuroprotective capacity [14,24–26] as well as having a great potential in preventing and reducing various types of cancer [8,27–33].

Although most of the articles mentioning the beneficial effects of walnuts are in vivo and in vitro experiments, new clinical studies describe the positive effects of walnut kernel consumption. In particular, they report that the long-term consumption of walnuts at ~15% of energy in the diet can reduce the concentrations of several inflammatory and metabolic syndrome biomarkers [34,35]; delay cognitive decline [36]; delay leukocyte telomere wear [37]; suppress breast cancer growth and survival [38]; reduce total mortality and CVD risk as well as increase life expectancy [39].

However, despite all these advantages, walnut oil, like all vegetable oils, can be unstable due to the sensitivity of its unsaturated fatty acids to oxidation. This oxidative sensitivity is regarded as one of the crucial parameters that indicate the quality, palatability, nutritional value, and toxicity of edible oils [40,41].

Several factors are cited as precursors of oil oxidation such as storage temperature, UV radiation, and oxygen in the air, which facilitate the oxidation of the unsaturated fatty acids by degrading their C–C double bonds, causing their transformation into secondary oxidation products such as ketones, aldehydes, and alcohols [40], and reducing their nutritional value [41]. On the other hand, roasting is one of the main pretreatments applied to oil seeds [42]. Different studies have shown the impact of roasting pretreatment on the composition of bioactive compounds such as phenols, sterols, and tocopherols as well as on the antioxidant activity and oxidative stability of oils [43–47].

This paper aimed to study the influence of both storage time at 60 °C (which can be considered as a stress condition and not a frying temperature and also the most used in various scientific papers with the same objective [48–51]) and traditional roasting on the physicochemical parameters, pigment content, and chemical composition as well as the antioxidant activity, in order to gain a better understanding of the stability behavior of walnut oils.

2. Materials and Methods

2.1. Plant Materials

The seeds of the *Juglans regia* plant studied in this paper were those from the Beni Mellal-Khenifra region—Morocco and, more precisely from the Azilal Province (31°58′00.0″ N, 6°34′00.0″ W), which is labelled as a geographical indication as “Azilal walnut”. After the fruits were harvested (September 2021), they were dried in the dark in a dry and ventilated area, peeled and then shelled manually to obtain only the kernels that were divided into two portions, one of which was treated with a traditional roasting process before the pressing stage and the other was directly cold-pressed. The extraction of oils was performed with the help of a cold press machine, Komet DD 85 G presses (IBG Monforts Oekotec GmbH, Mönchengladbach, Germany) to obtain two types of walnut oil: roasted walnut oil (RWO) and unroasted walnut oil (UWO). Samples of the collected oils were immediately analyzed, and the remainder were distributed in 30 mL non-transparent brown glass bottles well-filled to avoid the presence of air in contact with the oils. The bottles were placed in an oven (VWR, Sheldon manufacturing, INC. Cornelius, OR, USA) at a temperature of 60 ± 2 °C, and the analysis was repeated every 10 days in triplicate for two months.

2.2. Physicochemical Quality Parameters

The acid value (AV) was determined according to ISO 660 [52], the peroxide value was calculated in conformity with the ISO 3960 standard [53], while the specific extinction coefficient values (K_{232} and K_{270}), were determined according to the American Oil Chemists' Society (AOCS) recommended practice Ch 5-91 [54]. The acid value (AV) of an oil reflects the amount of potassium in mg required to neutralize its free acidity. The free acid content

of fats increases with time, and this index, therefore, makes it possible to judge their state of deterioration; this was measured in mg of KOH/g of oil. Coefficients of specific extinction at 232 and 270 nm were measured using a 1% (*w/v*) solution of the oil in cyclohexane in 1 cm cell way length, utilizing a UV spectrophotometer (Model UV-5800PC, manufactured by Shanghai Metash Instruments Co., Ltd., Shanghai, China).

2.3. Chlorophylls and Carotenoids Content

Chlorophyll and carotenoid determinations were performed by preparing a 1% solution of walnut seed oil in cyclohexane, followed by measuring absorbance at 670 nm and 470 nm, respectively [55]. The contents were calculated using the following formulas (Equations (1) and (2)).

$$\text{Chlorophyll (mg/kg)} = \frac{A_{670} \times 10^6}{613 \times 100 \times d} \quad (1)$$

$$\text{Carotenoid (mg/kg)} = \frac{A_{470} \times 10^6}{2000 \times 100 \times d} \quad (2)$$

The chlorophyll and carotenoid contents were reported in ppm (mg of predominant pheophytin per kilogram of oil) and mg of lutein per kilogram of oil, respectively.

2.4. Fatty Acid Composition

Saturated, monounsaturated, and polyunsaturated fatty acids were determined following the recommended practices required by the American Oil Chemists' Society (AOCS), Ce 1i-07 [54]. The fatty acids (FAs) undergo a transesterification reaction with KOH/methanol to convert into fatty acid methyl esters (FAMES), which were later analyzed by gas chromatography with a capillary column (Varian CP-3800, Varian Inc., Middelburg, The Netherlands). The equipment had a polar stationary phase and split inlet system, the separation was according to the fatty acid chain length (CL), degree of unsaturation, and position of double bonds (which means the position isomers). A fused silica column was used with the following dimensions ($l = 30 \text{ m}$, $\varnothing = 0.32 \text{ mm}$). The carrier gas used was helium for chromatography, and the initial and final column temperatures were $170 \text{ }^\circ\text{C}$ and $230 \text{ }^\circ\text{C}$, respectively, with a raising rate of $3 \text{ }^\circ\text{C}/\text{min}$. The injected volume was $1 \text{ } \mu\text{L}$, and the detection was with a FID detector at $230 \text{ }^\circ\text{C}$. The results were reported as percentages of each individual fatty acid present in the sample.

2.5. Tocopherol Composition

The amounts of tocopherol were quantified following the ISO 9936 standard method using an HPLC equipped with a fluorometric detector on a silica column ($25 \text{ cm} \times 4 \text{ mm}$). An isooctane:isopropanol (99%:1%) mixture was used as the eluent with a debit of $1.2 \text{ mL}/\text{min}$ for 20 min. Furthermore, the quantification was carried out with the help of standard external curves of α -, β -, γ -, and δ -tocopherols, and a quantitative and qualitative daily reference of tocopherol standards [56].

2.6. Phytosterols Composition

The sterol composition quantification was performed according to the Official AOCS Method Ch 6-91 [54], which requires saponification of each oil sample, then the extraction of the unsaponifiable fraction, followed by separation of the crude sterols by TLC, after the trimethylsilylation of the crude sterol fraction, and the determination of the sterol composition was performed by gas chromatography with a capillary column (Varian CP-3800, Varian Inc., Middelburg, The Netherlands). The column dimensions were $l = 30 \text{ m}$, $\varnothing = 0.32 \text{ mm}$) using helium as the carrier gas with an injection volume of $1 \text{ } \mu\text{L}$ for each analysis.

2.7. Radical Scavenging Activity

The radical scavenging activity of RWO and UWO was evaluated against the DPPH (2,2-diphenyl 1-picrylhydrazyl) free radical using a slightly modified Gharibzahedi method [57]. Concisely, 100 mg of each oil (RWO and UWO) in 1 mL of toluene was vortexed for 30 s with 3900 μ L of the DPPH toluene solution (10^{-4} M). The absorbance at 517 nm was measured after 30 min of mixing using a UV-Visible spectrophotometer (Model UV-5800PC UV/VIS Spectrophotometer, manufactured by Shanghai Metash Instruments Co. Ltd.). A mixture of 3900 μ L DPPH solution and 1100 μ L pure toluene was used as a negative control and the percentage of radical scavenging activity (%RSA) was calculated by Equation (3):

$$\%RSA = \frac{A_{\text{control}} - A_{\text{sample}}}{A_{\text{control}}} \times 100 \quad (3)$$

where A_{Control} is the absorbance of the negative control solution, and A_{Sample} is the absorbance of the sample solutions.

2.8. Statistical Analysis

GraphPad Prism 9 software was used to carry out the two-way ANOVA test for the verification of the statistical significance by Bonferroni's multiple comparisons test at a confidence level of 95.0%, and the results were expressed as the means \pm standard error of the mean of triplicate readings ($n = 3$). Differences at $p > 0.05$ were not considered statistically significant.

3. Results and Discussion

3.1. Physicochemical Quality Parameters, Chlorophylls, and Carotenoids Content

In Figure 1, we summarized the evolution of the contents of acid value, peroxide value, specific extinction coefficients (K_{232} and K_{270}), chlorophyll, and carotenoids present in the oils (RWO and UWO) for 60 days. We started with the acid value (AV) results, which illustrated the quality and edibility of the oils [58]. It clearly appears that on the first day, the roasting process increased the acid value in a significant way with values of 2.78 ± 0.04 and 2.27 ± 0.02 for RWO and UWO, respectively ($p < 0.0001$), and at the end of the storage period. These values increased for both the RWO and UWO with an AV of 5.84 ± 0.04 mg KOH/g of oil and 4.20 ± 0.10 mg KOH/g of oil, respectively (Figure 1A), which were significantly different ($p < 0.05$). It is interesting to note that even under these storage conditions, the acid value of RWO did not exceed 4.00 mg KOH/g of oil, which is the acceptable limit of the Codex Alimentarius [59], until 30 days, while UWO could withstand more than 50 days before losing its conformity. It can be said that the roasting process facilitates the elevation of the acid value over time, while roasted and unroasted walnut oils are more stable in terms of acid value than bitter and sweet almond oils, which showed higher values than ours under the same storage conditions over a shorter period [48].

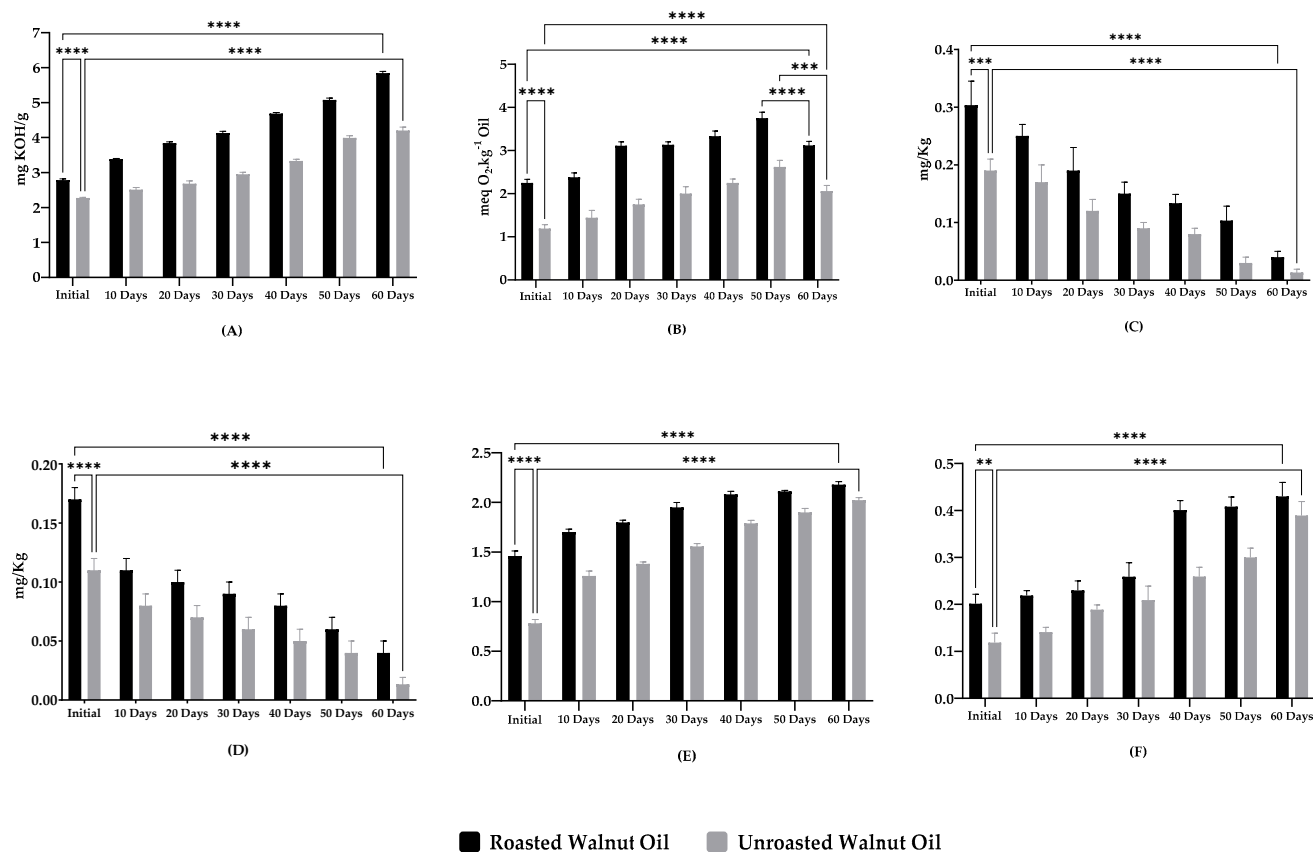


Figure 1. The physicochemical quality parameters, chlorophyll, and carotenoid content. (A) Evolution of acid value (mg KOH/g oil) during 60 days; (B) Evolution of peroxide value (meq $O_2 \cdot kg^{-1}$ oil) during 60 days; (C) Evolution of chlorophyll content (mg/kg) during 60 days; (D) Evolution of carotenoids content (mg/kg) during 60 days; (E) Evolution of K_{232} during 60 days; (F) Evolution of K_{270} during 60 days. ** (0.001 < p < 0.01); *** (0.0001 < p < 0.001); **** (p < 0.0001).

Concerning the peroxide value (PV), it represents one of the most widely used quality parameters to track the lipid oxidation and control the quality of the oil [60], providing a quantitative measure of the primary oxidation product levels in oils and fats [61]. According to the IOC (International Olive Council), 15 meq $O_2 \cdot kg^{-1}$ is the upper limit of acceptable oil quality [62]. Regarding the starting peroxide values, the UWO reached a value of 1.19 ± 0.09 meq $O_2 \cdot kg^{-1}$ while the RWO recorded a value of 2.25 ± 0.08 meq $O_2 \cdot kg^{-1}$, (Figure 1B) which means that the roasting process directly influences this parameter by increasing its value significantly ($p < 0.0001$). These values continued to increase until the 50th day to find the highest value with 2.62 ± 0.15 meq $O_2 \cdot kg^{-1}$ and 3.75 ± 0.14 meq $O_2 \cdot kg^{-1}$ for the UWO and RWO, respectively, and after that, they dropped to 2.06 ± 0.13 meq $O_2 \cdot kg^{-1}$ for UWO and 3.12 ± 0.09 meq $O_2 \cdot kg^{-1}$ for RWO ($p < 0.0001$). This decrease (between the 50 and 60 days for both RWO and UWO) can be explained by the fact that the peroxide index provides quantitative measurements on the primary oxidation products. It can be observed that the values increased from the first day until the 50th, which is the peak value of this index (PV). After that, the products of primary oxidation underwent a second oxidation to transform to volatile secondary oxidation products such as aldehydes [63], which are not detectable by this parameter (PV). For this particular reason, a decrease in the values in a significant way ($p < 0.0001$) was observed. Overall, the findings revealed that roasting and storage time influenced the PV results by increasing its values, but at the end of the study, RWO and UWO still showed conforming values according to the International Olive Council below the limit of 15 meq $O_2 \cdot kg^{-1}$.

Regarding the pigment dosage, it is clear that roasting favors the transfer of pigments to the oil (Figure 1C,D), and this is in line with the conclusions found by Adrián in 2018 [64].

The analysis by UV–Visible spectrophotometer detects higher pigment contents in RWO than in UWO ($p < 0.05$). Beginning with the determination of chlorophyll, which is a coloring substance that plays an important role in the antioxidant activity of vegetable oils [65]. The values recorded at the beginning of the test were 0.30 mg/kg and 0.19 mg/kg for the RWO and UWO, respectively ($p < 0.001$). However, these concentrations decreased progressively with time and dropped at the end of storage by more than 86% for the RWO and 94% of the initial value for the UWO ($p < 0.0001$), and this lowering of values may be sufficient to induce a slight rise in the oxidation rate of the oils [66]. Carotenoid molecules are also potent antioxidants [67], since they can scavenge singlet oxygen and provide various health benefits that include reducing the risk of chronic diseases such as degenerative eye and cardiovascular diseases [68–70]. Since carotenoids are highly unsaturated, they can degrade rapidly when stored under stressful conditions [71]. In this paper, carotenoids followed the same trend as chlorophyll, where RWO and UWO showed a loss at the end of the study of more than 76% and 90%, respectively ($p < 0.0001$), and these losses were justified by the high storage temperature associated with prolonged storage time, in line with what Agyare found in 2022 [72].

Figure 1E,F illustrate the absorption values at 232 nm (K_{232}) and 270 nm (K_{270}), respectively, which can be considered as an image of the oxidation state of vegetable oils, indicating the evolution of the formation of primary and secondary oxidation products [73]. A higher extinction value of 232 nm means that the product is more peroxidized. Likewise, the higher the extinction value at 270 nm, the richer in secondary oxidation products, and the worse its conservation properties [55]. It can be seen that roasting and storage time in the applied conditions directly impact the increase in the values of K_{232} and K_{270} , which signifies an increase in the oxidation level ($p < 0.05$) and a decrease in the quality of these oils. Starting with K_{232} and after 60 days of storage at 60 °C, the values increased from 1.46 ± 0.04 to 2.18 ± 0.03 for RWO and from 0.78 ± 0.04 to 2.03 ± 0.03 for UWO, which were significantly different ($p < 0.05$). This indicates an increase in the amount of primary oxidation product, but despite this increase, it remains within the acceptable limit for extra virgin olive oil, below the 2.5 required by the IOC [62,74]. On the other hand, K_{270} follows the same evolution as K_{232} with a duplication of the value from 0.20 ± 0.02 to 0.43 ± 0.03 for RWO ($p < 0.0001$), and a threefold increase for UWO from 0.11 ± 0.02 to 0.38 ± 0.03 ($p < 0.0001$). We noted that our results exceeded the value of 0.25 at the end of our investigation, which is the acceptable limit of extra virgin olive oil asked by the IOC [62]. This increase leads to roasted and unroasted walnut oils losing their conformity due to the transformation of the peroxides formed previously to volatile products such as ketones and aldehydes, which are known as secondary oxidation products [75,76].

3.2. Fatty Acid Composition

Juglans regia oil is considered as one of the most important oils in the human diet because of its essential fatty acid composition [77,78], specifically the mono- and polyunsaturated fatty acids that have several medicinal effects including the regulation of blood lipids and thrombus cleansing [79]. Changes in the fatty acid composition of roasted and unroasted walnut oils during 60 days of storage at 60 °C are reported in Table 1. In this study, the five main fatty acids in *Juglans regia* oil were compared: palmitic acid (C16:0), stearic acid (C18:0), oleic acid (C18:1), linoleic acid (C18:2), and linolenic acid (C18:3). Our findings showed a high percentage of polyunsaturated fatty acids (C18:2 and C18:3) exceeding 70%, 15% of monounsaturated fatty acids, and 13% of saturated fatty acids. The main unsaturated fatty acid in the studied oils was linoleic acid (C18:2) with percentages between $58.86 \pm 0.04\%$ and $60.99 \pm 0.03\%$ in the UWO ($p < 0.05$) and between $60.22 \pm 0.02\%$ and $60.43 \pm 0.02\%$ in the RWO ($p < 0.05$). In terms of saturated fatty acids, the major one is palmitic acid (C16:0) between $8.40 \pm 0.02\%$ and $9.44 \pm 0.02\%$ in UWO ($p < 0.05$) and between $8.57 \pm 0.03\%$ and $9.90 \pm 0.03\%$ in RWO ($p < 0.05$). Therefore, it can be said that statistically, the interaction between the roasting effect and storage time in the applied conditions affects the fatty acid composition. However, this impact remains minor, considering

that C18:2 is consistently the major fatty acid followed by C18:1, C18:3, C16:0, and C18:3 in accordance with the results found in different walnut cultivars [80,81] and in both the RWO and UWO oils during the whole period of this investigation.

Table 1. The fatty acid composition (%) of UWO and RWO during 60 days of storage at 60 ± 2 °C.

	C16:0	C18:0	C18:1	C18:2	C18:3	SFA/UFA
<i>Unroasted Walnut (Juglans regia L.) Seed Oils</i>						
Initial	9.90 ± 0.03 ^{1,a}	2.67 ± 0.05 ^{1,a}	14.65 ± 0.03 ^{1,a}	58.86 ± 0.04 ^{1,a}	13.42 ± 0.01 ^{1,a}	0.1
10	8.58 ± 0.01 ^{1,b}	2.41 ± 0.02 ^{1,b}	14.83 ± 0.02 ^{1,b}	60.23 ± 0.02 ^{1,b}	13.28 ± 0.02 ^{1,b}	0.1
20	8.61 ± 0.02 ^{1,b}	2.37 ± 0.02 ^{1,b}	14.61 ± 0.02 ^{1,a}	60.28 ± 0.01 ^{1,b,c}	13.52 ± 0.02 ^{1,c}	0.1
30	8.43 ± 0.03 ^{1,c}	2.34 ± 0.02 ^{1,b}	14.67 ± 0.03 ^{1,a}	60.48 ± 0.03 ^{1,d}	13.52 ± 0.01 ^{1,c}	0.1
40	8.23 ± 0.02 ^{1,d}	2.39 ± 0.01 ^{1,b}	14.77 ± 0.03 ^{1,b}	60.35 ± 0.04 ^{1,c}	13.66 ± 0.02 ^{1,d}	0.1
50	8.32 ± 0.03 ^{1,e}	2.38 ± 0.01 ^{1,b}	14.83 ± 0.02 ^{1,b}	60.50 ± 0.02 ^{1,d}	13.58 ± 0.03 ^{1,c,d}	0.1
60	8.57 ± 0.02 ^{1,b}	2.41 ± 0.02 ^{1,b}	15.01 ± 0.03 ^{1,c}	60.99 ± 0.03 ^{1,e}	12.54 ± 0.02 ^{1,e}	0.1
<i>Roasted Walnut (Juglans regia L.) Seed Oils</i>						
Initial	9.44 ± 0.02 ^{2,A}	2.32 ± 0.03 ^{2,A}	14.71 ± 0.03 ^{1,A}	60.22 ± 0.02 ^{2,A}	12.70 ± 0.04 ^{2,A}	0.1
10	8.64 ± 0.02 ^{1,B}	2.37 ± 0.02 ^{1,A,B}	14.50 ± 0.03 ^{2,B}	60.44 ± 0.01 ^{2,B}	13.39 ± 0.05 ^{2,B}	0.1
20	8.79 ± 0.03 ^{2,C}	2.31 ± 0.01 ^{1,A}	14.90 ± 0.03 ^{2,C}	60.47 ± 0.02 ^{2,B}	12.91 ± 0.03 ^{2,C}	0.1
30	8.94 ± 0.02 ^{2,D}	2.36 ± 0.02 ^{1,A,B}	15.21 ± 0.02 ^{2,D}	59.55 ± 0.02 ^{2,C}	13.37 ± 0.02 ^{2,B}	0.1
40	8.52 ± 0.01 ^{2,E}	2.40 ± 0.01 ^{1,B}	14.55 ± 0.01 ^{2,B,E}	60.32 ± 0.01 ^{1,D}	13.60 ± 0.02 ^{1,D}	0.1
50	8.49 ± 0.01 ^{2,E}	2.33 ± 0.02 ^{1,A,B,C}	14.60 ± 0.02 ^{2,E}	60.49 ± 0.01 ^{1,B}	13.55 ± 0.03 ^{1,D,E}	0.1
60	8.40 ± 0.02 ^{2,F}	2.41 ± 0.03 ^{1,B}	14.58 ± 0.01 ^{2,E}	60.43 ± 0.02 ^{1,B}	13.51 ± 0.01 ^{2,E}	0.1

Data with the same superscript numbers in the same column for roasting in the same period were not significantly different ($p > 0.05$). Data with the same lowercase letters in the same column for time of storage of UWO were not significantly different ($p > 0.05$). Data with the same uppercase letters in the same column for time of storage of RWO were not significantly different ($p > 0.05$).

3.3. Tocopherol Composition

In recent years, antioxidant molecules have attracted considerable interest and among these, molecules stand out the tocopherols, which are known as one of the most essential antioxidants in nutrition due to their liposolubility [82] and their various biological activities [83]. These include the prevention of various types of cancer [84–90] and cardiovascular disease [91–93], and all these benefits have led us to be interested in the composition and evolution of these tocopherol molecules and all their isomers. Table 2 shows the contents of α , γ , and δ -tocopherol as well as the total tocopherol values in mg/kg. It is noteworthy that β -tocopherol was not detected in the *Juglans regia* oils or is present in trace amounts, in accordance with what was found by Cecilia Cittadini in 2020 for Argentinian walnut oil [94]. γ -Tocopherol was detected as the primary tocopherol in both RWO and UWO, followed by δ and α -tocopherol, respectively. In terms of the values of γ -tocopherol, it ranged between 606.32 ± 1.80 mg/kg and 508.68 ± 3.20 mg/kg in UWO ($p < 0.05$) and between 624.77 ± 2.30 mg/kg and 579.56 ± 2.20 mg/kg in RWO ($p < 0.05$). These results are higher than those recorded by Bou Abdallah for six different Tunisian walnut cultivars [95]. Regarding the contents of total tocopherols, our results showed that at the beginning and without the effects of storage time, the contents of the total tocopherols increased from 669.45 ± 2.50 mg/kg in UWO to 680.50 ± 2.05 mg/kg in RWO ($p < 0.05$). Similarly, at the end of this study, we recorded values of 639.80 ± 1.45 mg/kg in UWO and 665.24 ± 1.10 mg/kg in RWO ($p < 0.05$). This indicated that roasting had a positive effect on increasing the total tocopherol content, which can be explained by the fact that during roasting, there is damage at the membrane, which leads to the release of a more significant amount of these molecules (tocopherols) in oil [96,97]. This subsequently explains the increase in the antioxidant activity of roasted seed oils compared to the unroasted ones. According to the effect of the storage time at 60 °C, the results showed that the oil retained almost the same content of total tocopherols with a loss of 4.43% for UWO and 2.24% for RWO, which means that the studied oils are stable and can resist in such storage conditions.

Table 2. The tocopherol composition (mg/kg) of UWO and RWO during 60 days of storage at 60 ± 2 °C.

	α -Tocopherol	γ -Tocopherol	δ -Tocopherol	Total
<i>Unroasted Walnut (Juglans regia L.) Seed Oils</i>				
Initial	9.37 ± 0.08 ^{1,a}	606.32 ± 1.80 ^{1,a}	50.74 ± 0.93 ^{1,a}	669.45 ± 2.50 ^{1,a}
10	7.25 ± 0.10 ^{1,b}	609.02 ± 2.40 ^{1,a}	47.28 ± 1.65 ^{1,a}	665.01 ± 3.33 ^{1,a,b}
20	9.47 ± 0.06 ^{1,a,d}	604.54 ± 1.90 ^{1,a}	48.57 ± 1.54 ^{1,a}	662.58 ± 2.13 ^{1,b}
30	10.97 ± 0.13 ^{1,c}	605.28 ± 1.60 ^{1,a}	44.61 ± 0.90 ^{1,b}	660.86 ± 1.96 ^{1,b,c}
40	9.76 ± 0.05 ^{1,d}	597.71 ± 0.90 ^{1,b}	44.95 ± 1.05 ^{1,b}	655.24 ± 2.85 ^{1,c}
50	9.03 ± 0.05 ^{1,e}	584.93 ± 2.80 ^{1,c}	48.77 ± 0.85 ^{1,a,c}	645.12 ± 3.09 ^{1,d}
60	8.88 ± 0.11 ^{1,e}	580.68 ± 3.20 ^{1,c}	49.91 ± 1.30 ^{1,c}	639.80 ± 1.45 ^{1,d}
				Loss (%) = 4.43
<i>Roasted Walnut (Juglans regia L.) Seed Oils</i>				
Initial	9.60 ± 0.20 ^{1,A}	624.77 ± 2.30 ^{2,A}	46.07 ± 0.75 ^{2,A}	680.50 ± 2.05 ^{2,A}
10	8.19 ± 0.13 ^{2,B}	621.70 ± 1.90 ^{2,A}	46.83 ± 1.20 ^{1,A}	676.72 ± 1.60 ^{2,A,B}
20	7.47 ± 0.05 ^{2,C}	619.66 ± 0.93 ^{2,A}	45.68 ± 1.05 ^{1,A,B}	672.81 ± 0.95 ^{2,B,C}
30	9.16 ± 0.08 ^{2,D}	611.76 ± 1.50 ^{2,B}	47.45 ± 0.80 ^{1,A}	668.37 ± 0.88 ^{2,C}
40	8.65 ± 0.11 ^{2,E}	605.73 ± 2.50 ^{1,B}	44.84 ± 1.44 ^{1,A}	660.41 ± 0.53 ^{1,D}
50	10.35 ± 0.12 ^{2,F}	598.34 ± 1.70 ^{2,C}	45.38 ± 0.43 ^{1,A,B}	662.67 ± 0.90 ^{2,C,D}
60	11.97 ± 0.09 ^{2,G}	579.56 ± 2.20 ^{1,D}	42.38 ± 0.94 ^{2,B}	665.24 ± 1.10 ^{2,C,D}
				Loss (%) = 2.24

Data with the same superscript numbers in the same column for roasting in the same period were not significantly different ($p > 0.05$). Data with the same lowercase letters in the same column for time of storage of UWO were not significantly different ($p > 0.05$). Data with the same uppercase letters in the same column for time of storage of RWO were not significantly different ($p > 0.05$).

3.4. Sterol Composition

Walnut (*Juglans regia*) oil is a highly recommended oil due to its richness in phytosterol, which has the capacity to prevent cardiovascular diseases [48,98]. These molecules are more hydrophobic than cholesterol and can dislodge cholesterol from intestinal micelles and reduce LDL absorption. In combination with linolenic acid, phytosterols have both complementary and synergistic lipid-lowering effects [35], and also has an impressive antioxidant activity [99–101]. The individual and total sterol composition of UWO and RWO during the different storage times under the applied conditions are summarized in Table 3. The most predominant phytosterol is β -sitosterol, followed by Δ^5 -avenasterol and campesterol, confirmed by various compositional studies of walnut oil [102–104]. During storage at 60 °C, the amount of the significant phytosterols, β -sitosterol, decreased with losses of 13.78% in UWO, from 1058.09 mg/kg to 912.23 mg/kg and 17.49% in RWO, from 1056.98 mg/kg to 872.07 mg/kg. The same goes for the total sterol contents, which also dropped by 15.06% in UWO, from 1260.38 mg/kg to 1070.57 mg/kg, and by 17.42% in RWO from 1236.66 mg/kg to 1021.28 mg/kg. All of these results were significantly different at $p < 0.05$. Therefore, we can affirm that our storage conditions negatively impacted on the phytosterol content, similar to what was previously found in the sweet and bitter almond oil study [48]. Roasting also had a negative influence on individual and total sterols, similar to what was found previously in a study on sesame oil [105], which justifies this by the oxidation of these compounds at high roasting temperatures. This is in contrast to another study that found a higher level in roasted flaxseed oil compared to the unroasted ones. We can attribute these results to the cell structure damage created during the roasting process, which subsequently leads to an enhancement in the extractability of phytosterols [106]. However, it is important to note that even under these storage conditions (60 °C for 60 days) and the roasting process, walnut oils (UWO and RWO) maintained their phytosterol content with a slight decrease.

Table 3. The sterol composition (mg/kg) of UWO and RWO during 60 days of storage at 60 ± 2 °C.

	Cholesterol	Campesterol	Stigmasterol	β -Sitosterol	$\Delta 5$ -Avenasterol	$\Delta 7$ -Stigmasterol	$\Delta 7$ -Avenasterol	Total Sterols
<i>Unroasted Walnut (Juglans regia L.) Seed Oils</i>								
Initial	26.47 \pm 0.03 ^{1,a}	64.28 \pm 0.15 ^{1,a}	3.53 \pm 0.001 ^{1,a}	1058.09 \pm 1.02 ^{1,a}	70.58 \pm 0.07 ^{1,a}	0.63 \pm 0.001 ^{1,a}	1.51 \pm 0.001 ^{1,a}	1260.38 \pm 1.22 ^{1,a}
10 Days	5.13 \pm 0.01 ^{1,b}	62.75 \pm 0.07 ^{1,b}	4.64 \pm 0.001 ^{1,b}	1048.85 \pm 1.13 ^{1,b}	72.52 \pm 0.08 ^{1,b}	4.40 \pm 0.001 ^{1,b}	1.10 \pm 0.001 ^{1,b}	1220.87 \pm 1.31 ^{1,b}
20 Days	13.25 \pm 0.02 ^{1,c}	59.21 \pm 0.07 ^{1,c}	9.26 \pm 0.01 ^{1,c}	993.02 \pm 1.19 ^{1,c}	59.09 \pm 0.07 ^{1,c}	1.03 \pm 0.001 ^{1,c}	5.39 \pm 0.01 ^{1,c}	1172.4 \pm 1.40 ^{1,c}
30 Days	29.05 \pm 0.04 ^{1,d}	64.21 \pm 0.09 ^{1,a}	11.46 \pm 0.02 ^{1,d}	957.01 \pm 1.40 ^{1,d}	65.04 \pm 0.09 ^{1,d}	1.14 \pm 0.001 ^{1,d}	2.88 \pm 0.001 ^{1,d}	1150.51 \pm 1.68 ^{1,d}
40 Days	14.69 \pm 0.01 ^{1,e}	59.84 \pm 0.05 ^{1,d}	7.50 \pm 0.01 ^{1,e}	912.11 \pm 0.81 ^{1,e}	53.86 \pm 0.05 ^{1,e}	2.34 \pm 0.001 ^{1,e}	1.78 \pm 0.001 ^{1,e}	1072.39 \pm 0.95 ^{1,e}
50 Days	13.53 \pm 0.01 ^{1,f}	60.36 \pm 0.06 ^{1,e}	6.61 \pm 0.01 ^{1,f}	919.54 \pm 0.90 ^{1,f}	53.80 \pm 0.05 ^{1,e}	1.97 \pm 0.001 ^{1,f}	1.51 \pm 0.001 ^{1,a}	1070.09 \pm 1.05 ^{1,e}
60 Days	14.67 \pm 0.02 ^{1,e}	58.02 \pm 0.07 ^{1,f}	3.32 \pm 0.01 ^{1,g}	912.23 \pm 1.14 ^{1,e}	60.81 \pm 0.07 ^{1,f}	0.95 \pm 0.001 ^{1,g}	1.43 \pm 0.001 ^{1,f}	1070.57 \pm 1.33 ^{1,e}
<i>Roasted Walnut (Juglans regia L.) Seed Oils</i>								
Initial	6.06 \pm 0.01 ^{2,A}	59.98 \pm 0.06 ^{2,A}	4.82 \pm 0.01 ^{2,A}	1056.98 \pm 1.10 ^{1,A}	69.38 \pm 0.07 ^{2,A}	12.24 \pm 0.01 ^{2,A}	1.24 \pm 0.001 ^{2,A}	1236.66 \pm 1.29 ^{2,A}
10 Days	14.10 \pm 0.01 ^{2,B}	59.00 \pm 0.06 ^{2,B}	2.96 \pm 0.001 ^{2,B}	1013.16 \pm 0.95 ^{2,B}	55.44 \pm 0.05 ^{2,B}	2.13 \pm 0.001 ^{2,B}	0.24 \pm 0.001 ^{2,B}	1184.7 \pm 1.11 ^{2,B}
20 Days	10.09 \pm 0.01 ^{2,C}	56.56 \pm 0.06 ^{2,C}	2.99 \pm 0.001 ^{2,C}	977.70 \pm 1.05 ^{2,C}	62.89 \pm 0.07 ^{2,B}	0.97 \pm 0.001 ^{2,C}	1.25 \pm 0.001 ^{2,C}	1132.7 \pm 1.21 ^{2,C}
30 Days	16.68 \pm 0.02 ^{2,D}	54.44 \pm 0.07 ^{2,D}	4.28 \pm 0.01 ^{2,D}	937.00 \pm 1.17 ^{2,D}	62.88 \pm 0.08 ^{2,C}	0.10 \pm 0.001 ^{2,D}	0.91 \pm 0.001 ^{2,D}	1097.32 \pm 1.37 ^{2,D}
40 Days	9.56 \pm 0.02 ^{2,E}	53.80 \pm 0.07 ^{2,E}	2.94 \pm 0.001 ^{2,B}	902.25 \pm 1.23 ^{2,E}	60.94 \pm 0.08 ^{2,D}	0.95 \pm 0.001 ^{2,E}	1.37 \pm 0.001 ^{2,E}	1050.72 \pm 1.43 ^{2,E}
50 Days	14.17 \pm 0.02 ^{2,F}	58.58 \pm 0.06 ^{2,F}	2.26 \pm 0.001 ^{2,E}	891.68 \pm 0.97 ^{2,F}	59.59 \pm 0.06 ^{2,E}	0.83 \pm 0.001 ^{2,F}	1.27 \pm 0.001 ^{2,F}	1038.51 \pm 1.13 ^{2,F}
60 Days	15.52 \pm 0.01 ^{2,G}	50.66 \pm 0.02 ^{2,G}	3.98 \pm 0.001 ^{2,F}	872.07 \pm 0.43 ^{2,G}	58.52 \pm 0.03 ^{2,F}	0.09 \pm 0.001 ^{2,G}	0.84 \pm 0.001 ^{2,G}	1021.28 \pm 0.50 ^{2,G}

Data with the same superscript numbers in the same column for roasting in the same period were not significantly different ($p > 0.05$). Data with the same lowercase letters in the same column for time of storage of UWO were not significantly different ($p > 0.05$). Data with the same uppercase letters in the same column for time of storage of RWO were not significantly different ($p > 0.05$).

3.5. Antioxidant Activity

In this study, changes in the spectrometric absorbance were used to plot the histogram of radical scavenging activity evolution (Figure 2). However, our results showed that walnut oil had a very high antioxidant capacity in accordance with the literature [107–110], with RSA percentages above 60% for all of the samples. Initial values of %RSA were equal to $86.15 \pm 1.08\%$ for UWO and $94.81 \pm 1.20\%$ for RWO, which were significantly different ($p < 0.0001$). Contrary to what we expected, it can be easily concluded that, initially, roasting has a direct impact on antioxidant activity by increasing the power and capacity of the oil to scavenge the DPPH free radicals. These results can be explained by the fact that during the roasting process, Maillard reaction products (MRPs) are formed after the initiation of chemical reactions between reducing sugars and amino acids present in the oils [111,112], which leads to a significant enhancement in the antioxidant capacity [113,114]. However, this activity will decrease with time, under the influence of the temperature applied during the storage period (two months) with a loss of 17% for RWO and 13% for UWO at the end of the study ($p < 0.0001$). This may be due to the considerable content of tocopherols (vitamin E) in *Juglans regia* oil [115–117], which acts as an essential antioxidant that protects the oils from degrading and losing their properties [118].

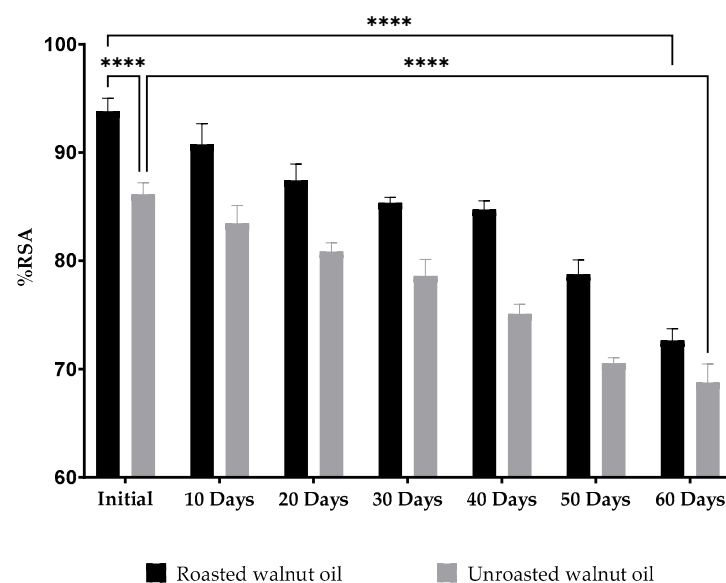


Figure 2. The evolution of the radical scavenging activity (RSA) during 60 days. **** ($p < 0.0001$).

4. Conclusions

The effects of roasting and accelerated storage conditions were investigated in this study. Acid value, peroxide value, and specific extinction (K_{232} and K_{270}) increased with the roasting and storage time. Roasting enhanced the pigment transfer into the oil, which increased the chlorophyll and carotenoid values, while these values decreased with storage time. The total sterol content slightly reduced, influenced by both factors applied in this study. While the total content of tocopherols was increased by the roasting process, this increased content of tocopherols was strongly correlated with the antioxidant activity, which leads to an increase in the percentage of the radical scavenging activity (%RSA) of walnut oil against the DPPH radical. However, all of the parameters evaluated in this article were negatively affected by the storage conditions applied. At the same time, the roasting process has shown an important role in increasing the vitamin E content and the antioxidant capacity, preventing oxidation and degradation of the investigated oils, and increasing its nutritional value. In spite of all the valuable information provided in this article, further research should be considered as perspectives to further valorize these oils, among these, a study on the stability of walnut seed oil under the influence of different roasting temperatures as well as the effect of UV radiation on the photodegradation of bioactive compounds in walnut seeds oils, which are currently in progress. However, other studies are still needed to valorize Moroccan walnut oils in the pharmacological field by evaluating the antioxidant activity against a wide range of free radicals (DPPH, ABTS, FRAP, ORAC, etc.) as well as the antimicrobial, antifungal, and antidiabetic activities of these oils. This research will improve the understanding of the stability behavior of cold-pressed walnut oils, which will facilitate their preservation and storage as well as their applications in various nutritional and pharmacological fields.

Author Contributions: Conceptualization, A.B.; Data curation, Y.E. and A.E.Y.; Formal analysis, Y.E., Z.L.E.I., A.A.A.A. and A.B.; Funding acquisition, A.E.Y. and K.W.G.; Investigation, Z.L.E.I. and M.M.A.; Methodology, Z.L.E.I., M.M.A. and M.T.; Project administration, A.E.Y., L.C.M. and A.B.; Resources, Y.E. and L.C.M.; Software, K.W.G.; Supervision, M.T.; Validation, H.H., K.W.G. and M.T.; Writing—original draft, Y.E. and A.B.; Writing—review & editing, H.H., A.A.A.A., K.W.G. and L.C.M. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: All data were included in this manuscript.

Conflicts of Interest: The authors declare no conflict of interest.

References

1. Catanzaro, E.; Greco, G.; Potenza, L.; Calcabrini, C.; Fimognari, C. Natural Products to Fight Cancer: A Focus on *Juglans Regia*. *Toxins* **2018**, *10*, 469. [[CrossRef](#)] [[PubMed](#)]
2. Shigaeva, J.; Darr, D. On the Socio-Economic Importance of Natural and Planted Walnut (*Juglans regia* L.) Forests in the Silk Road Countries: A Systematic Review. *Policy Econ.* **2020**, *118*, 102233. [[CrossRef](#)]
3. Uddin, Y.; Khan, N.M.; Ali, F.; Ahamd, S.; Khan, Z.U.; Nawaz, M.A.; Wang, J. Estimation of Various Physicochemical Properties of Walnut Oil from Different Areas of Northern Kpk, Pakistan. *J. Mex. Chem. Soc.* **2021**, *65*, 572–581. [[CrossRef](#)]
4. Liu, M.; Li, C.; Cao, C.; Wang, L.; Li, X.; Che, J.; Yang, H.; Zhang, X.; Zhao, H.; He, G.; et al. Walnut Fruit Processing Equipment: Academic Insights and Perspectives. *Food Eng. Rev.* **2021**, *13*, 822–857. [[CrossRef](#)]
5. Nguyen, T.H.D.; Vu, D.C. A Review on Phytochemical Composition and Potential Health-Promoting Properties of Walnuts. *Food Rev. Int.* **2021**, *10*, 1–27. [[CrossRef](#)]
6. Rusu, M.E.; Georgiu, C.; Pop, A.; Mocan, A.; Kiss, B.; Vostinaru, O.; Fizesan, I.; Stefan, M.-G.; Gheldiu, A.-M.; Mates, L.; et al. Antioxidant Effects of Walnut (*Juglans regia* L.) Kernel and Walnut Septum Extract in a D-Galactose-Induced Aging Model and in Naturally Aged Rats. *Antioxidants* **2020**, *9*, 424. [[CrossRef](#)]
7. Alasalvar, C.; Bolling, B.W. Review of Nut Phytochemicals, Fat-Soluble Bioactives, Antioxidant Components and Health Effects. *Br. J. Nutr.* **2015**, *113*, S68–S78. [[CrossRef](#)]

8. Reiter, R.J.; Tan, D.-X.; Manchester, L.C.; Korkmaz, A.; Fuentes-Broto, L.; Hardman, W.E.; Rosales-Corral, S.A.; Qi, W. A Walnut-Enriched Diet Reduces the Growth of LNCaP Human Prostate Cancer Xenografts in Nude Mice. *Cancer Invest.* **2013**, *31*, 365–373. [[CrossRef](#)]
9. Soussi, A.; Gargouri, M.; el Feki, A. Potential Immunomodulatory and Antioxidant Effects of Walnut *Juglans regia* Vegetable Oil against Lead-Mediated Hepatic Damage and Their Interaction with Lipase Activity in Rats. *Environ. Toxicol.* **2018**, *33*, 1261–1271. [[CrossRef](#)]
10. Bati, B.; Celik, I.; Dogan, A. Determination of Hepatoprotective and Antioxidant Role of Walnuts Against Ethanol-Induced Oxidative Stress in Rats. *Cell Biochem. Biophys.* **2015**, *71*, 1191–1198. [[CrossRef](#)]
11. Qamar, W.; Sultana, S. Polyphenols from *Juglans regia* L. (Walnut) Kernel Modulate Cigarette Smoke Extract Induced Acute Inflammation, Oxidative Stress and Lung Injury in Wistar Rats. *Hum. Exp. Toxicol.* **2011**, *30*, 499–506. [[CrossRef](#)] [[PubMed](#)]
12. Fukuda, T.; Ito, H.; Yoshida, T. Effect of the Walnut Polyphenol Fraction on Oxidative Stress in Type 2 Diabetes Mice. *BioFactors* **2004**, *21*, 251–253. [[CrossRef](#)] [[PubMed](#)]
13. Tsoukas, M.A.; Ko, B.-J.; Witte, T.R.; Dincer, F.; Hardman, W.E.; Mantzoros, C.S. Dietary Walnut Suppression of Colorectal Cancer in Mice: Mediation by miRNA Patterns and Fatty Acid Incorporation. *J. Nutr. Biochem.* **2015**, *26*, 776–783. [[CrossRef](#)] [[PubMed](#)]
14. Poulou, S.M.; Bielinski, D.F.; Shukitt-Hale, B. Walnut Diet Reduces Accumulation of Polyubiquitinated Proteins and Inflammation in the Brain of Aged Rats. *J. Nutr. Biochem.* **2013**, *24*, 912–919. [[CrossRef](#)] [[PubMed](#)]
15. Thangthaeng, N.; Poulou, S.M.; Fisher, D.R.; Shukitt-Hale, B. Walnut Extract Modulates Activation of Microglia through Alteration in Intracellular Calcium Concentration. *Nutr. Res.* **2018**, *49*, 88–95. [[CrossRef](#)]
16. Willis, L.M.; Bielinski, D.F.; Fisher, D.R.; Matthan, N.R.; Joseph, J.A. Walnut Extract Inhibits LPS-Induced Activation of Bv-2 Microglia via Internalization of TLR4: Possible Involvement of Phospholipase D2. *Inflammation* **2010**, *33*, 325–333. [[CrossRef](#)]
17. Koh, S.-J.; Choi, Y.-I.; Kim, Y.; Kim, Y.-S.; Choi, S.W.; Kim, J.W.; Kim, B.G.; Lee, K.L. Walnut Phenolic Extract Inhibits Nuclear Factor KappaB Signaling in Intestinal Epithelial Cells, and Ameliorates Experimental Colitis and Colitis-Associated Colon Cancer in Mice. *Eur. J. Nutr.* **2019**, *58*, 1603–1613. [[CrossRef](#)]
18. Laubertová, L.; Koňariková, K.; Gbelcová, H.; Ďuračková, Z.; Žitňanová, I. Effect of Walnut Oil on Hyperglycemia-Induced Oxidative Stress and pro-Inflammatory Cytokines Production. *Eur. J. Nutr.* **2015**, *54*, 291–299. [[CrossRef](#)]
19. Papoutsis, Z.; Kassi, E.; Chinou, I.; Halabalaki, M.; Skaltsounis, L.A.; Moutsatsou, P. Walnut Extract (*Juglans regia* L.) and Its Component Ellagic Acid Exhibit Anti-Inflammatory Activity in Human Aorta Endothelial Cells and Osteoblastic Activity in the Cell Line KS483. *Br. J. Nutr.* **2008**, *99*, 715–722. [[CrossRef](#)]
20. Beigh, S.; Rashid, H.; Sharma, S.; Parvez, S.; Raisuddin, S. Bleomycin-Induced Pulmonary Toxicopathological Changes in Rats and Its Prevention by Walnut Extract. *Biomed. Pharmacother.* **2017**, *94*, 418–429. [[CrossRef](#)]
21. Ho, K.-V.; Lei, Z.; Sumner, L.; Coggeshall, M.; Hsieh, H.-Y.; Stewart, G.; Lin, C.-H. Identifying Antibacterial Compounds in Black Walnuts (*Juglans nigra*) Using a Metabolomics Approach. *Metabolites* **2018**, *8*, 58. [[CrossRef](#)] [[PubMed](#)]
22. Pereira, J.A.; Oliveira, I.; Sousa, A.; Ferreira, I.C.F.R.; Bento, A.; Estevinho, L. Bioactive Properties and Chemical Composition of Six Walnut (*Juglans regia* L.) Cultivars. *Food Chem. Toxicol.* **2008**, *46*, 2103–2111. [[CrossRef](#)] [[PubMed](#)]
23. Amarowicz, R.; Dykes, G.A.; Pegg, R.B. Antibacterial Activity of Tannin Constituents from *Phaseolus vulgaris*, *Fagopyrum esculentum*, *Corylus avellana* and *Juglans nigra*. *Fitoterapia* **2008**, *79*, 217–219. [[CrossRef](#)] [[PubMed](#)]
24. Haider, S.; Batool, Z.; Ahmad, S.; Siddiqui, R.A.; Haleem, D.J. Walnut Supplementation Reverses the Scopolamine-Induced Memory Impairment by Restoration of Cholinergic Function via Mitigating Oxidative Stress in Rats: A Potential Therapeutic Intervention for Age Related Neurodegenerative Disorders. *Metab. Brain Dis.* **2018**, *33*, 39–51. [[CrossRef](#)]
25. Choi, J.; Park, G.; Kim, H.; Oh, D.-S.; Kim, H.; Oh, M. In Vitro and in Vivo Neuroprotective Effects of Walnut (*Juglans regia* L.) in Models of Parkinson's Disease. *Int. J. Mol. Sci.* **2016**, *17*, 108. [[CrossRef](#)]
26. Essa, M.M.; Subash, S.; Dhanalakshmi, C.; Manivasagam, T.; Al-Adawi, S.; Guillemain, G.J.; Thenmozhi, A.J. Dietary Supplementation of Walnut Partially Reverses 1-Methyl-4-Phenyl-1,2,3,6-Tetrahydropyridine Induced Neurodegeneration in a Mouse Model of Parkinson's Disease. *Neurochem. Res.* **2015**, *40*, 1283–1293. [[CrossRef](#)]
27. Khir, R.; Pan, Z. Walnuts. In *Integrated Processing Technologies for Food and Agricultural by-Products*; Elsevier: Amsterdam, The Netherlands, 2019; pp. 391–411.
28. Bourais, I.; Elmarrakechy, S.; Taha, D.; Mourabit, Y.; Bouyahya, A.; el Yadini, M.; Machich, O.; el Hajjaji, S.; el Boury, H.; Dakka, N.; et al. A Review on Medicinal Uses, Nutritional Value, and Antimicrobial, Antioxidant, Anti-Inflammatory, Antidiabetic, and Anticancer Potential Related to Bioactive Compounds of *J. Regia*. *Food Rev. Int.* **2022**, *1*–51. [[CrossRef](#)]
29. Davis, P.A.; Vasu, V.T.; Gohil, K.; Kim, H.; Khan, I.H.; Cross, C.E.; Yokoyama, W. A High-Fat Diet Containing Whole Walnuts (*Juglans regia*) Reduces Tumour Size and Growth along with Plasma Insulin-like Growth Factor 1 in the Transgenic Adenocarcinoma of the Mouse Prostate Model. *Br. J. Nutr.* **2012**, *108*, 1764–1772. [[CrossRef](#)]
30. Schlörmann, W.; Lamberty, J.; Ludwig, D.; Lorkowski, S.; Gleib, M. In Vitro–Fermented Raw and Roasted Walnuts Induce Expression of CAT and GSTT2 Genes, Growth Inhibition, and Apoptosis in LT97 Colon Adenoma Cells. *Nutr. Res.* **2017**, *47*, 72–80. [[CrossRef](#)]
31. Chung, J.; Kim, Y.-S.; Lee, J.; Lee, J.H.; Choi, S.-W.; Kim, Y. Compositional Analysis of Walnut Lipid Extracts and Properties as an Anti-Cancer Stem Cell Regulator via Suppression of the Self-Renewal Capacity. *Food Sci. Biotechnol.* **2016**, *25*, 623–629. [[CrossRef](#)]
32. Nagel, J.M.; Brinkoetter, M.; Magkos, F.; Liu, X.; Chamberland, J.P.; Shah, S.; Zhou, J.; Blackburn, G.; Mantzoros, C.S. Dietary Walnuts Inhibit Colorectal Cancer Growth in Mice by Suppressing Angiogenesis. *Nutrition* **2012**, *28*, 67–75. [[CrossRef](#)] [[PubMed](#)]

33. Lee, J.; Kim, Y.S.; Lee, J.H.; Heo, S.C.; Lee, K.L.; Choi, S.W.; Kim, Y. Walnut Phenolic Extract and Its Bioactive Compounds Suppress Colon Cancer Cell Growth by Regulating Colon Cancer Stemness. *Nutrients* **2016**, *8*, 439. [CrossRef] [PubMed]
34. Cofán, M.; Rajaram, S.; Sala-Vila, A.; Valls-Pedret, C.; Serra-Mir, M.; Roth, I.; Freitas-Simoes, T.M.; Bitok, E.; Sabaté, J.; Ros, E. Effects of 2-Year Walnut-Supplemented Diet on Inflammatory Biomarkers. *J. Am. Coll. Cardiol.* **2020**, *76*, 2282–2284. [CrossRef]
35. Mateş, L.; Popa, D.S.; Rusu, M.E.; Fizeşan, I.; Leucuta, D. Walnut Intake Interventions Targeting Biomarkers of Metabolic Syndrome and Inflammation in Middle-Aged and Older Adults: A Systematic Review and Meta-Analysis of Randomized Controlled Trials. *Antioxidants* **2022**, *11*, 1412. [CrossRef] [PubMed]
36. Sala-Vila, A.; Valls-Pedret, C.; Rajaram, S.; Coll-Adrós, N.; Cofán, M.; Serra-Mir, M.; Pérez-Heras, A.M.; Roth, I.; Freitas-Simoes, T.M.; Doménech, M.; et al. Effect of a 2-Year Diet Intervention with Walnuts on Cognitive Decline. The Walnuts And Healthy Aging (WAHA) Study: A Randomized Controlled Trial. *Am. J. Clin. Nutr.* **2020**, *111*, 590–600. [CrossRef]
37. Freitas-Simoes, T.M.; Cofán, M.; Blasco, M.A.; Soberón, N.; Foronda, M.; Serra-Mir, M.; Roth, I.; Valls-Pedret, C.; Doménech, M.; Ponferrada-Ariza, E.; et al. Walnut Consumption for Two Years and Leukocyte Telomere Attrition in Mediterranean Elders: Results of a Randomized Controlled Trial. *Nutrients* **2018**, *10*, 1907. [CrossRef]
38. Hardman, W.E.; Primerano, D.A.; Legenza, M.T.; Morgan, J.; Fan, J.; Denvir, J. Dietary Walnut Altered Gene Expressions Related to Tumor Growth, Survival, and Metastasis in Breast Cancer Patients: A Pilot Clinical Trial. *Nutr. Res.* **2019**, *66*, 82–94. [CrossRef]
39. Liu, X.; Guasch-Ferré, M.; Tobias, D.K.; Li, Y. Association of Walnut Consumption with Total and Cause-Specific Mortality and Life Expectancy in U.S. Adults. *Nutrients* **2021**, *13*, 2699. [CrossRef]
40. Morales, M.T.; Przybylski, R. Olive Oil Oxidation. In *Handbook of Olive Oil: Analysis and Properties*; Springer: Berlin, Germany, 2013; pp. 479–522. [CrossRef]
41. Piergiovanni, L.; Limbo, S. Packaging and the Shelf Life of Vegetable Oils. In *Food Packaging and Shelf Life*; CRC Press: New York, NY, USA, 2009; pp. 317–338.
42. Durmaz, G.; Gökmen, V. Changes in Oxidative Stability, Antioxidant Capacity and Phytochemical Composition of Pistacia Terebinthus Oil with Roasting. *Food Chem.* **2011**, *128*, 410–414. [CrossRef]
43. Suri, K.; Singh, B.; Kaur, A.; Yadav, M.P.; Singh, N. Impact of Infrared and Dry Air Roasting on the Oxidative Stability, Fatty Acid Composition, Maillard Reaction Products and Other Chemical Properties of Black Cumin (*Nigella sativa* L.) Seed Oil. *Food. Chem.* **2019**, *295*, 537–547. [CrossRef]
44. Suri, K.; Singh, B.; Kaur, A.; Singh, N. Impact of Roasting and Extraction Methods on Chemical Properties, Oxidative Stability and Maillard Reaction Products of Peanut Oils. *J. Food Sci. Technol.* **2019**, *56*, 2436–2445. [CrossRef] [PubMed]
45. al Juhaimi, F.; Musa Özcan, M.; Ghafoor, K.; Babiker, E.E. The Effect of Microwave Roasting on Bioactive Compounds, Antioxidant Activity and Fatty Acid Composition of Apricot Kernel and Oils. *Food. Chem.* **2018**, *243*, 414–419. [CrossRef] [PubMed]
46. Ghafoor, K.; Özcan, M.M.; AL-Juhaimi, F.; Babiker, E.E.; Fadimu, G.J. Changes in Quality, Bioactive Compounds, Fatty Acids, Tocopherols, and Phenolic Composition in Oven- and Microwave-Roasted Poppy Seeds and Oil. *LWT* **2019**, *99*, 490–496. [CrossRef]
47. Suri, K.; Singh, B.; Kaur, A.; Yadav, M.P.; Singh, N. Influence of Microwave Roasting on Chemical Composition, Oxidative Stability and Fatty Acid Composition of Flaxseed (*Linum usitatissimum* L.) Oil. *Food. Chem.* **2020**, *326*, 126974. [CrossRef]
48. el Bernoussi, S.; Boujemaa, I.; Harhar, H.; Belmaghraoui, W.; Matthäus, B.; Tabyaoui, M. Evaluation of Oxidative Stability of Sweet and Bitter Almond Oils under Accelerated Storage Conditions. *J. Stored Prod. Res.* **2020**, *88*, 101662. [CrossRef]
49. Choe, E.; Min, D.B. Mechanisms and Factors for Edible Oil Oxidation. *Compr. Rev. Food. Sci. Food. Saf.* **2006**, *5*, 169–186. [CrossRef]
50. Morsy, M.K.; Sami, R.; Algarni, E.; Al-Mushhin, A.A.M.; Benajiba, N.; Almasoudi, A.; Almasoudi, A.G.; Mekawi, E. Phytochemical Profile and Antioxidant Activity of Sesame Seed (*Sesamum indicum*) By-Products for Stability and Shelf Life Improvement of Refined Olive Oil. *Antioxidants* **2022**, *11*, 338. [CrossRef]
51. Ghafoor, K.; al Juhaimi, F.; Özcan, M.M.; Ahmed, I.A.M.; Babiker, E.E.; Alsawmahi, O.N. Evaluation of the Antioxidant Activity of Some Plant Extracts (Rosemary, Sage, and Savory, Summer) on Stability of Moringa Oil. *J. Food Process Preserv.* **2021**, *45*, e15203. [CrossRef]
52. ISO 660. *Animal and Vegetable Fats and Oils—Determination of Acid Value and Acidity*, 4th ed.; 2020; Volume 2020, Available online: <https://www.iso.org/standard/75594.html> (accessed on 25 May 2022).
53. ISO 3960. *Animal and Vegetable Fats and Oils—Determination of Peroxide Value—Iodometric (Visual) Endpoint Determination*, 5th ed.; 2017; Available online: <https://www.iso.org/standard/71268.html> (accessed on 25 May 2022).
54. AOCS. *Official Methods and Recommended Practices of the American Oil Chemists' Society*, 7th ed.; 2017; Available online: [https://www.scirp.org/\(S\(351jmbntv-nsjt1aadkpozje\)\)/reference/referencespapers.aspx?referenceid=3019699](https://www.scirp.org/(S(351jmbntv-nsjt1aadkpozje))/reference/referencespapers.aspx?referenceid=3019699) (accessed on 25 May 2022).
55. Wolff, J.P. *Manuel d'analyse Des Corps Gras*; Azoulay éd: Paris, France, 1968.
56. ISO 9936. *Animal and Vegetable Fats and Oils—Determination of Tocopherol and Tocotrienol Contents by High-Performance Liquid Chromatography*; 2016; Volume 2016, Available online: <https://www.iso.org/standard/69595.html> (accessed on 25 May 2022).
57. Siger, A.; Nogala-Kalucka, M.; Lampart-Szczapa, E. The content and antioxidant activity of phenolic compounds in cold-pressed plant oils. *J. Food Lipids* **2008**, *15*, 137–149. [CrossRef]
58. Boujemaa, I.; el Bernoussi, S.; Harhar, H.; Tabyaoui, M. The Influence of the Species on the Quality, Chemical Composition and Antioxidant Activity of Pumpkin Seed Oil. *OCL-Oilseeds Fats Crops Lipids* **2020**, *27*, 2020031. [CrossRef]

59. Codex Alimentarius. *FAO/WHO Food Standards Programme Codex Alimentarius Commission*, 42nd ed.; 2019; pp. 2–7. Available online: <https://www.fao.org/3/ca2329en/ca2329en.pdf> (accessed on 25 May 2022).
60. Zhang, N.; Li, Y.; Wen, S.; Sun, Y.; Chen, J.; Gao, Y.; Sagymbek, A.; Yu, X. Analytical Methods for Determining the Peroxide Value of Edible Oils: A Mini-Review. *Food Chem* **2021**, *358*, 129834. [[CrossRef](#)] [[PubMed](#)]
61. Grilo, F.S.; Wang, S.C. Walnut (*Juglans regia* L.) Volatile Compounds Indicate Kernel and Oil Oxidation. *Foods* **2021**, *10*, 329. [[CrossRef](#)] [[PubMed](#)]
62. International Olive Council. *Commercial Standard Applicable to Olive Oils and Olive-Pomace Oils*; IOC/T.15/NC N°3/Rev 14; 2019; Available online: <https://www.internationaloliveoil.org/wp-content/uploads/2019/12/trade-standard-REV-14-Eng.pdf> (accessed on 25 May 2022).
63. Grebenteuch, S.; Kroh, L.W.; Drusch, S.; Rohn, S. Formation of Secondary and Tertiary Volatile Compounds Resulting from the Lipid Oxidation of Rapeseed Oil. *Foods* **2021**, *10*, 2417. [[CrossRef](#)] [[PubMed](#)]
64. Rabadán, A.; Gallardo-Guerrero, L.; Gandul-Rojas, B.; Álvarez-Ortí, M.; Pardo, J.E. Effect of Roasting Conditions on Pigment Composition and Some Quality Parameters of Pistachio Oil. *Food. Chem.* **2018**, *264*, 49–57. [[CrossRef](#)] [[PubMed](#)]
65. Pérez-Gálvez, A.; Viera, I.; Roca, M. Carotenoids and Chlorophylls as Antioxidants. *Antioxidants* **2020**, *9*, 505. [[CrossRef](#)]
66. Crowe, T.D.; White, P.J. Oxidative Stability of Walnut Oils Extracted with Supercritical Carbon Dioxide. *J. Am. Oil Chem. Soc.* **2003**, *80*, 575–578. [[CrossRef](#)]
67. Marc, F.; Davin, A.; Deglène-Benbrahim, L.; Ferrand, C.; Baccaunaud, M.; Fritsch, P. Méthodes d'évaluation Du Potentiel Antioxydant Dans Les Aliments. *Médecine Sci.* **2004**, *20*, 387–490. [[CrossRef](#)]
68. Wiesner, M.; Hanschen, F.S.; Maul, R.; Neugart, S.; Schreiner, M.; Baldermann, S. Nutritional Quality of Plants for Food and Fodder. In *Encyclopedia of Applied Plant Sciences*; Elsevier: Amsterdam, The Netherlands, 2017; Volume 1, pp. 285–291.
69. Stevenson, D.G.; Eller, F.J.; Wang, L.; Jane, J.L.; Wang, T.; Inglett, G.E. Oil and Tocopherol Content and Composition of Pumpkin Seed Oil in 12 Cultivars. *J. Agric. Food Chem.* **2007**, *55*, 4005–4013. [[CrossRef](#)]
70. KOPSELL, D.; KOPSELL, D. Accumulation and Bioavailability of Dietary Carotenoids in Vegetable Crops. *Trends Plant Sci.* **2006**, *11*, 499–507. [[CrossRef](#)]
71. Rodriguez-Amaya, D.B. Changes in Carotenoids during Processing and Storage of Foods. *Arch. Latinoam. Nutr.* **1999**, *49*, 38S–47S.
72. Agyare, A.N.; An, C.H.; Liang, Q. Goji Berry (*Lycium barbarum* L.) Carotenoids Enrichment through 'Green' Extraction Method Improves Oxidative Stability and Maintains Fatty Acids of Yak Ghee with Microwave Heating and Storage. *Foods* **2022**, *11*, 369. [[CrossRef](#)] [[PubMed](#)]
73. El Mouddeh, H.; El Idrissi, Y.; El Guezane, C.; Belmaghraoui, W.; El Yadini, A.; Harhar, H.; Tabyaoui, M. Tradition Mills' Picholine Olive Oil Physicochemical Characterization and Chemical Profiling across Different Cities in Morocco. *Sci. World J.* **2020**, *2020*, 1804723. [[CrossRef](#)] [[PubMed](#)]
74. Isabel Minguez-Mosquera, M.; Rejano-Navarro, L.; Gandul-Rojas, B.; SanchezGomez, A.H.; Garrido-Fernandez, J. Color-pigment Correlation in Virgin Olive Oil. *J. Am. Oil Chem. Soc.* **1991**, *68*, 332–336. [[CrossRef](#)]
75. Allouche, Y.; Jiménez, A.; Gaforio, J.J.; Uceda, M.; Beltrán, G. How Heating Affects Extra Virgin Olive Oil Quality Indexes and Chemical Composition. *J. Agric. Food Chem.* **2007**, *55*, 9646–9654. [[CrossRef](#)] [[PubMed](#)]
76. Conte, L.; Milani, A.; Calligaris, S.; Rovellini, P.; Lucci, P.; Nicoli, M.C. Temperature Dependence of Oxidation Kinetics of Extra Virgin Olive Oil (EVOO) and Shelf-Life Prediction. *Foods* **2020**, *9*, 295. [[CrossRef](#)] [[PubMed](#)]
77. Geng, S.; Ning, D.; Ma, T.; Chen, H.; Zhang, Y.; Sun, X. Comprehensive Analysis of the Components of Walnut Kernel (*Juglans regia* L.) in China. *J. Food Qual.* **2021**, *2021*, 9302181. [[CrossRef](#)]
78. Binici, H.İ.; Şat, İ.; Aoudeh, E. Nutritional Composition and Health Benefits of Walnut and Its Products. *Atatürk Üniversitesi Ziraat Fakültesi Derg.* **2021**, *52*, 224–230. [[CrossRef](#)]
79. Gao, P.; Jin, J.; Liu, R.; Jin, Q.; Wang, X. Chemical Compositions of Walnut (*Juglans regia* L.) Oils from Different Cultivated Regions in China. *J. Am. Oil Chem. Soc.* **2018**, *95*, 825–834. [[CrossRef](#)]
80. Amaral, J.S.; Casal, S.; Pereira, J.A.; Seabra, R.M.; Oliveira, B.P.P. 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.* **2003**, *51*, 7698–7702. [[CrossRef](#)]
81. Gao, P.; Liu, R.; Jin, Q.; Wang, X. Comparative Study of Chemical Compositions and Antioxidant Capacities of Oils Obtained from Two Species of Walnut: *Juglans regia* and *Juglans sigillata*. *Food. Chem.* **2019**, *279*, 279–287. [[CrossRef](#)]
82. Shahidi, F.; de Camargo, A. Tocopherols and Tocotrienols in Common and Emerging Dietary Sources: Occurrence, Applications, and Health Benefits. *Int. J. Mol. Sci.* **2016**, *17*, 1745. [[CrossRef](#)]
83. Zingg, J.-M. Modulation of Signal Transduction by Vitamin E. *Mol. Aspects Med.* **2007**, *28*, 481–506. [[CrossRef](#)] [[PubMed](#)]
84. Kalender, Y.; Yel, M.; Kalender, S. Doxorubicin Hepatotoxicity and Hepatic Free Radical Metabolism in Rats. *Toxicology* **2005**, *209*, 39–45. [[CrossRef](#)] [[PubMed](#)]
85. Hu, F.; Wu, Z.; Li, G.; Teng, C.; Liu, Y.; Wang, F.; Zhao, Y.; Pang, D. The Plasma Level of Retinol, Vitamins A, C and α -Tocopherol Could Reduce Breast Cancer Risk? A Meta-Analysis and Meta-Regression. *J. Cancer Res. Clin. Oncol.* **2015**, *141*, 601–614. [[CrossRef](#)] [[PubMed](#)]
86. Tam, K.-W.; Ho, C.-T.; Lee, W.-J.; Tu, S.-H.; Huang, C.-S.; Chen, C.-S.; Lee, C.-H.; Wu, C.-H.; Ho, Y.-S. Alteration of α -Tocopherol-Associated Protein (TAP) Expression in Human Breast Epithelial Cells during Breast Cancer Development. *Food. Chem.* **2013**, *138*, 1015–1021. [[CrossRef](#)]

87. Wada, S.; Satomi, Y.; Murakoshi, M.; Noguchi, N.; Yoshikawa, T.; Nishino, H. Tumor Suppressive Effects of Tocotrienol in Vivo and in Vitro. *Cancer Lett.* **2005**, *229*, 181–191. [[CrossRef](#)]
88. Yu, W.; Simmons-Menchaca, M.; Gapor, A.; Sanders, B.G.; Kline, K. Induction of Apoptosis in Human Breast Cancer Cells by Tocopherols and Tocotrienols. *Nutr. Cancer* **1999**, *33*, 26–32. [[CrossRef](#)]
89. Virtamo, J.; Taylor, P.R.; Kontto, J.; Männistö, S.; Utriainen, M.; Weinstein, S.J.; Huttunen, J.; Albanes, D. Effects of A-tocopherol and B-carotene Supplementation on Cancer Incidence and Mortality: 18-Year Postintervention Follow-up of the Alpha-Tocopherol, Beta-Carotene Cancer Prevention Study. *Int. J. Cancer* **2014**, *135*, 178–185. [[CrossRef](#)]
90. Kong, P.; Cai, Q.; Geng, Q.; Wang, J.; Lan, Y.; Zhan, Y.; Xu, D. Vitamin Intake Reduce the Risk of Gastric Cancer: Meta-Analysis and Systematic Review of Randomized and Observational Studies. *PLoS ONE* **2014**, *9*, e116060. [[CrossRef](#)]
91. Jialal, I.; Fuller, C.J.; Huet, B.A. The Effect of α -Tocopherol Supplementation on LDL Oxidation. *Arter. Thromb. Vasc. Biol.* **1995**, *15*, 190–198. [[CrossRef](#)]
92. Loffredo, L.; Perri, L.; di Castelnuovo, A.; Iacoviello, L.; de Gaetano, G.; Violi, F. Supplementation with Vitamin E Alone Is Associated with Reduced Myocardial Infarction: A Meta-Analysis. *Nutr. Metab. Cardiovasc. Dis.* **2015**, *25*, 354–363. [[CrossRef](#)] [[PubMed](#)]
93. Christen, S.; Woodall, A.A.; Shigenaga, M.K.; Southwell-Keely, P.T.; Duncan, M.W.; Ames, B.N. γ -Tocopherol Traps Mutagenic Electrophiles Such as NO_x and Complements α -Tocopherol: Physiological Implications. *Proc. Natl. Acad. Sci. USA* **1997**, *94*, 3217–3222. [[CrossRef](#)] [[PubMed](#)]
94. Cittadini, M.C.; Martín, D.; Gallo, S.; Fuente, G.; Bodoira, R.; Martínez, M.; Maestri, D. 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.* **2020**, *246*, 833–843. [[CrossRef](#)]
95. Abdallah, I.B.; Tlili, N.; Martinez-Force, E.; Rubio, A.G.P.; Perez-Camino, M.C.; Albouchi, A.; Boukhchina, S. Content of Carotenoids, Tocopherols, Sterols, Triterpenic and Aliphatic Alcohols, and Volatile Compounds in Six Walnuts (*Juglans regia* L.) Varieties. *Food Chem.* **2015**, *173*, 972–978. [[CrossRef](#)] [[PubMed](#)]
96. Vaidya, B.; Choe, E. Effects of Seed Roasting on Tocopherols, Carotenoids, and Oxidation in Mustard Seed Oil During Heating. *J. Am. Oil Chem. Soc.* **2011**, *88*, 83–90. [[CrossRef](#)]
97. Lee, Y. Chemical Composition and Oxidative Stability of Safflower Oil Prepared from Safflower Seed Roasted with Different Temperatures. *Food Chem.* **2004**, *84*, 1–6. [[CrossRef](#)]
98. de Jong, A.; Plat, J.; Mensink, R.P. Metabolic Effects of Plant Sterols and Stanols (Review). *J. Nutr. Biochem.* **2003**, *14*, 362–369. [[CrossRef](#)]
99. Rodríguez-Yoldi, M.J. Anti-Inflammatory and Antioxidant Properties of Plant Extracts. *Antioxidants* **2021**, *10*, 921. [[CrossRef](#)]
100. Hussain, S.Z.; Naseer, B.; Qadri, T.; Fatima, T.; Bhat, T.A. *Fruits Grown in Highland Regions of the Himalayas*; Springer International Publishing: Cham, Switzerland, 2021; ISBN 978-3-030-75501-0.
101. Kreis, S.; Arendt, E.K.; Hübner, F.; Zarnkov, M. Cereal-Based Gluten-Free Functional Drinks. In *Gluten-Free Cereal Products and Beverages*; Elsevier: Amsterdam, The Netherlands, 2008; pp. 373–392.
102. Martínez, M.L.; Labuckas, D.O.; Lamarque, A.L.; Maestri, D.M. Walnut (*Juglans regia* L.): Genetic Resources, Chemistry, by-Products. *J. Sci. Food Agric* **2010**, *90*, 1959–1967. [[CrossRef](#)]
103. Bada, J.C.; León-Camacho, M.; Prieto, M.; Copovi, P.; Alonso, L. Characterization of Walnut Oils (*Juglans regia* L.) from Asturias, Spain. *J. Am. Oil Chem. Soc.* **2010**, *87*, 1469–1474. [[CrossRef](#)]
104. Rébua, C.; Artaud, J.; le Dréau, Y. Walnut (*Juglans regia* L.) Oil Chemical Composition Depending on Variety, Locality, Extraction Process and Storage Conditions: A Comprehensive Review. *J. Food Compos. Anal.* **2022**, *110*, 104534. [[CrossRef](#)]
105. Arab, R.; Casal, S.; Pinho, T.; Cruz, R.; Freidja, M.L.; Lorenzo, J.M.; Hano, C.; Madani, K.; Boulekbache-Makhlouf, L. Effects of Seed Roasting Temperature on Sesame Oil Fatty Acid Composition, Lignan, Sterol and Tocopherol Contents, Oxidative Stability and Antioxidant Potential for Food Applications. *Molecules* **2022**, *27*, 4508. [[CrossRef](#)] [[PubMed](#)]
106. Waszkowiak, K.; Siger, A.; Rudzińska, M.; Bamber, W. Effect of Roasting on Flaxseed Oil Quality and Stability. *J. Am. Oil Chem. Soc.* **2020**, *97*, 637–649. [[CrossRef](#)]
107. Grosso, A.L.; Asensio, C.M.; Nepote, V.; Grosso, N.R. Antioxidant Activity Displayed by Phenolic Compounds Obtained from Walnut Oil Cake Used for Walnut Oil Preservation. *J. Am. Oil Chem. Soc.* **2018**, *95*, 1409–1419. [[CrossRef](#)]
108. Copolovici, D.; Bungau, S.; Boscencu, R.; Tit, D.M.; Copolovici, L. The Fatty Acids Composition and Antioxidant Activity of Walnut Cold Press Oil. *Rev. De Chim.* **2017**, *68*, 507–509. [[CrossRef](#)]
109. Gao, P.; Ding, Y.; Chen, Z.; Zhou, Z.; Zhong, W.; Hu, C.; He, D.; Wang, X. Characteristics and Antioxidant Activity of Walnut Oil Using Various Pretreatment and Processing Technologies. *Foods* **2022**, *11*, 1698. [[CrossRef](#)]
110. Ahmed, I.A.M.; Al-Juhaimi, F.Y.; Özcan, M.M.; Osman, M.A.; Gassem, M.A.; Salih, H.A.A. 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.* **2019**, *68*, 167–173. [[CrossRef](#)]
111. Chandrasekara, N.; Shahidi, F. Effect of Roasting on Phenolic Content and Antioxidant Activities of Whole Cashew Nuts, Kernels, and Testa. *J. Agric. Food. Chem.* **2011**, *59*, 5006–5014. [[CrossRef](#)]
112. el Guezane, C.; el Moudden, H.; Harhar, H.; Warad, I.; Bellaouchou, A.; Guenbour, A.; Zarrouk, A.; Tabyaoui, M. Optimization of Roasting Conditions on the Bioactive Compounds and Their Antioxidant Power from *Opuntia Ficus-Indica* Seeds Using Response Surface Methodology (RSM). *Biointerface Res. Appl. Chem.* **2020**, *11*, 10510–10532. [[CrossRef](#)]

113. Ni, Z.-J.; Liu, X.; Xia, B.; Hu, L.-T.; Thakur, K.; Wei, Z.-J. Effects of Sugars on the Flavor and Antioxidant Properties of the Maillard Reaction Products of Camellia Seed Meals. *Food. Chem. X* **2021**, *11*, 100127. [[CrossRef](#)]
114. Nooshkam, M.; Varidi, M.; Bashash, M. The Maillard Reaction Products as Food-Born Antioxidant and Antibrowning Agents in Model and Real Food Systems. *Food. Chem.* **2019**, *275*, 644–660. [[CrossRef](#)] [[PubMed](#)]
115. Jahanban-Esfahlan, A.; Ostadrahimi, A.; Tabibiazar, M.; Amarowicz, R. A Comparative Review on the Extraction, Antioxidant Content and Antioxidant Potential of Different Parts of Walnut (*Juglans regia* L.) Fruit and Tree. *Molecules* **2019**, *24*, 2133. [[CrossRef](#)] [[PubMed](#)]
116. Yang, H.; Xiao, X.; Li, J.; Wang, F.; Mi, J.; Shi, Y.; He, F.; Chen, L.; Zhang, F.; Wan, X. Chemical Compositions of Walnut (*Juglans* spp.) Oil: Combined Effects of Genetic and Climatic Factors. *Forests* **2022**, *13*, 962. [[CrossRef](#)]
117. Gao, P.; Cao, Y.; Liu, R.; Jin, Q.; Wang, X. Phytochemical Content, Minor-Constituent Compositions, and Antioxidant Capacity of Screw-Pressed Walnut Oil Obtained from Roasted Kernels. *Eur. J. Lipid Sci. Technol.* **2019**, *121*, 1800292. [[CrossRef](#)]
118. Niki, E.; Noguchi, N. Antioxidant Action of Vitamin E in Vivo as Assessed from Its Reaction Products with Multiple Biological Oxidants. *Free Radic. Res.* **2021**, *55*, 352–363. [[CrossRef](#)]