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Comparative analyses of three dehydration methods on drying characteristics and oil quality of in-shell walnuts

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ABSTRACT

Developing an effective drying method for in-shell walnuts (*Juglans regia* L.) is a major postharvest processing concern in the nut industry. Three drying methods, including hot air drying (AD), vacuum drying (VD), and hot air-assisted radio frequency drying (ARFD), were experimentally compared and analyzed. The changes in lipid oxidation attributes, fatty acid composition, total antioxidant capacity (TAC), and total phenolic concentration (TPC) of walnuts were determined after dehydration and during storage. The results showed that the drying time required for in-shell walnuts using ARFD was the shortest (138 min), followed by VD (185 min) and AD required the longest time (300 min). Particularly, AD resulted in the highest lipid oxidation, followed by VD and ARFD. The walnuts treated by ARFD contained more unsaturated fatty acid than those treated by AD. Moreover, both the reduced power assay and free radical scavenging capacity tests showed that ARFD and VD had little effect on the TAC and TPC of walnuts during the drying process and storage. Overall, ARFD provides an effective and rapid drying method for in-shell walnuts.

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Antioxidant activity; hot air; lipid oxidation; radio frequency; storage; vacuum

Introduction

Walnuts (*Juglans regia* L.) are widely distributed tree nuts and rank second after almonds in global nut production. The world production of in-shell walnuts was about 4.146 Mt in 2015/2016 and China is the major walnut producer, accounting for approximately 50% of the total world production.[1] In the past decade, the worldwide production of walnuts doubled, undoubtedly reflecting an increasing demand for such nuts. Walnut kernels are nutrient-rich and an excellent source of antioxidant products mainly owing to their high levels of oil (54%, on average, unsaturated fatty acids (UFAs)), high total phenolic concentration (TPC), and total antioxidant capacity (TAC).[2] Many studies have verified the substantial beneficial impacts of walnut antioxidant compounds on human health.[3,4] However, walnuts are susceptible to germination and decay due to their high water and oil contents at harvest, which would result in poor quality stability and short shelf life of walnut products.[5] Drying of walnuts immediately after harvest is critical to preserve their quality. Fresh walnuts must be dried to a moisture content of 8.0 and 5.0% on a dry weight basis (d.b.) of the whole walnuts and kernels, respectively, for long time storage in an ambient environment.[6]

Several studies have investigated the effects of different drying methods, including microwave, convective, vacuum, and freeze-drying, on the drying characteristics and product quality of carrot,[7] raisins,[8] collard leaves,[9] rice,[10], and seaweed[11]. Unfortunately, reports on the effects of conventional and radio frequency (RF) drying on the quality of walnuts, especially the lipid quality and antioxidant activity of walnuts, are still limited. It is, therefore, necessary to compare different drying methods in terms of walnut quality and design the optimal and effective drying treatment protocol for in-shell walnuts.

Traditionally, walnut drying is performed using a hot air drying (AD) method in industrial applications and characterized by low energy efficiency and very long drying times. This is probably because loss of moisture often leads to a poor heat conduction in outer layers, resulting in very low water transfer during AD.[12] Moreover, the rate of heat transfer from the surface into the interior material is low due to poor thermal conduction, which eventually reduces the drying rate

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(DR). In addition, the heat and mass transfers occur in completely opposite directions. Thus, long exposure to relatively high drying temperatures frequently leads to undesirable thermal degradation in the finished products.[13]

Vacuum drying (VD) is an effective method of dehydration especially for products that are heat sensitive. Vacuum reduces the boiling point of water in products, which allows drying at lower temperature and reduces quality deterioration. During VD, water molecules absorb high energy and diffuse to the product surface. The vacuum in chamber reduces water vapor concentration at the surface of materials, hence creating a large vapor pressure gradient between the center and the surface, leading to rapid drying. Furthermore, low concentration of oxygen should reduce the oxidative rancidity or other forms of lipid oxidation. Therefore, both low drying temperature and reduced oxygen during drying improve final product quality, such as color, rehydration, and texture.[14] However, VD has some disadvantages. For example, it is difficult to deliver thermal energy to the products because convection heat transfer is absent in VD. In addition, VD has very high operating costs, mainly owing to the need to maintain vacuum during the entire drying periods.[15]

Radio frequency heating is a subset of several electromagnetic-based heating methods in which RF energy interacts with dielectric materials to generate heat as a result of transforming electromagnetic energy to thermal one. RF heating takes place at molecular and atomic levels as a result of polar and ionic polarizations in dielectric materials when exposed to electromagnetic waves. Dielectric heating with RF energy has demonstrated practical applications in baking, thawing, pasteurizing, and disinfesting.[16–18]

Radio frequency drying (RFD), referred to as a fourth-generation drying technology,[13] has some advantages over conventional drying, including shorter drying time, higher energy efficiency, and less quality deterioration. Several nuts have been successfully dried using RF energy, such as macadamia nuts[19] and in-shell walnuts.[20] However, the effects of RFD on the product quality, especially the total phenolics and antioxidant activities of walnuts, have not been reported.

The general objective of this study was to experimentally analyze and compare the effects of hot air drying, vacuum drying, and hot air-assisted radio frequency drying (ARFD) on the drying characteristics and product quality of in-shell walnuts with regard to acid value (AV), peroxide value (PV), saponification value (SV), iodine value (IV), fatty acid compositions, TPC, and TAC. This study also evaluated the storage stability of oils extracted from dried walnuts during an accelerated shelf life experiment.

Materials and methods

Materials

Walnuts (*J. regia* L.) with green husks were harvested in the mountain areas in Yangling, Shaanxi, China, in mid September, 2016. All walnuts used for drying were obtained from the same batch. To ensure the uniform size of samples used in the experiment, the walnuts were prescreened before being transported to the laboratory. Immediately after arriving, the walnuts were hulled and then washed with clean tap water. After cleaning, the nuts were directly placed on the ground in the sun, for about 10 h per day, and the whole process was completed in 2 d at a mean temperature of 25°C, yielding a kernel moisture content of about 20% d.b. Finally, the samples were packed and vacuum-sealed into aluminum bags. All packaged samples were stored in a refrigerator at 4°C until utilized for experiments.

Before experiments, vacuum packaged samples were placed in an incubator (BSC-150, Boxun Industry & Commerce Co., Ltd, Shanghai, China) at 25°C and 20% relative humidity overnight to obtain a uniform temperature (25 ± 0.5°C).

Drying equipment

A temperature-adjustable hot air-dryer (DG100D, Zhongkong Lab equipment Inc, Zhejiang, China) equipped with steam flow tray was used for AD experiments. A vacuum oven (DZX-6020B, Nanrong Lab equipment Inc, Shanghai, China) with adjustable vacuum pressure was used for VD experiments.

A 6 kW, 27.12 MHz pilot-scale free running oscillator RF system (SO6B, Strayfield International, Wokingham, UK) combined with a hot-air system supplied by a 6 kW electric heater was used for ARFD experiments (Figure 1). The dimension of the top electrode was

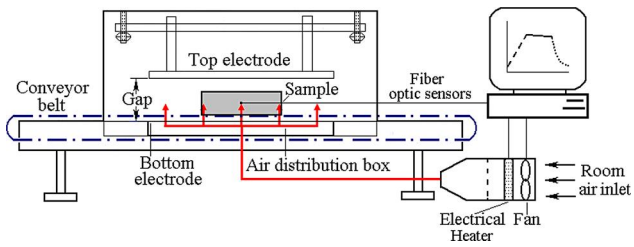

Figure 1. Schematic view of the pilot-scale 6 kW, 27.12 MHz RF system showing the plate electrodes, conveyor belt, and the hot air system (adapted from Wang et al.[19]).

71 × 52 cm. The detailed description of the RF unit and hot-air heating system can be found in Wang et al.[21]

Drying methods

Hot air drying: 20 in-shell walnut samples (178.52 g) were spread uniformly as a single layer in a plastic container (20 cm *L* × 16 cm *W* × 5 cm *H*) made of polyvinyl chloride with perforated side and bottom walls and placed in the cavity center of the tray dryer. The temperature and relative humidity of hot air were set to 70°C and 20%, respectively, with an air velocity of 2.0 m/s measured by a rotating vane anemometer (LCA 6000, AIRFLOW Instrumentation, Buckingham-215 Shire, UK). The whole in-shell walnut samples were dehydrated until they reached the final moisture content (8.0%, d.b.). The temperature of walnut kernels subjected to AD was measured at six positions in the container (Figure 2) every 20 min using a type T thermocouple (0.8 mm diameter and 0.8 s response time). The sensors were inserted through predrilled holes in the walnut shell. The walnuts were subsequently weighed using an electronic balance (PTX-FA210, Huazhi Scientific Instrument, Co., Ltd., Fuzhou, China) with a precision of 0.01 g to record the loss of water during the drying period.

Vacuum drying: 20 samples (182.54 g) were spread into thin layers in the container on the plate of vacuum chamber in which the temperature was also set at 70°C, and vacuum pressure was adjusted to −0.07 MPa[22] to control the water boiling point below 70°C. During the experiment, the vacuum was interrupted temporarily when the container was taken out of the vacuum chamber every 15 min. Walnut kernel temperature at six positions in the container (Figure 2), as described above, was simultaneously measured using type T thermocouples, and the samples were then weighed using the electronic balance. All the measurements were completed within 0.5 min. The container was placed back into the vacuum chamber again for continued drying until the water content of whole in-shell walnut decreased to 8.0% d.b.

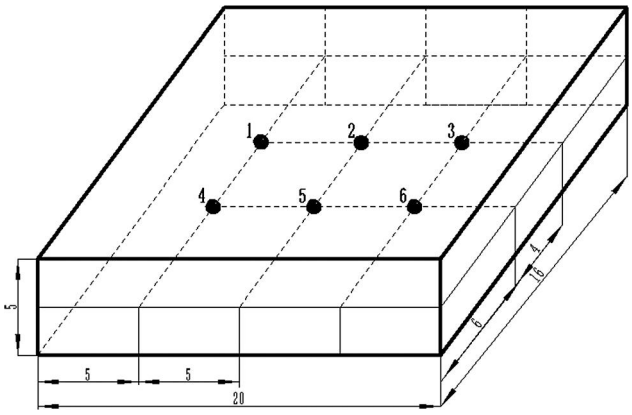

Figure 2. Rectangular plastic container with six locations for sample temperature measurements (all dimension are in cm).

Hot air-assisted radio frequency drying: 20 in-shell walnuts (174.63 g) were also placed in the same type of plastic container. Zhang et al.[17] previously found that after an initial warm-up period during ARFD, the average temperature of walnut kernels reached their highest value and then remained at a fairly constant temperature because the absorbed RF energy was balanced by the latent heat and sensible heat of water. They found that an electrode gap of 18.0 cm combined with a hot air temperature of 50°C provided an acceptable heating uniformity and stable sample temperature (70°C) during the RF drying process. According to these earlier studies, an RF drying process using an electrode gap of 18.0 cm combined with 50°C hot air was selected for further comparative tests. To monitor the temperature profile of walnut kernels during RF drying periods, six fiber-optic temperature sensors (HQ-FTS-D120, Heqi Technologies Inc., Xian, China) were inserted into the center of walnut kernels at six positions within the container (Figure 2) described previously. In addition, the container was removed from the RF system through the infeed side every 10 min to record the weight of the samples. The RF drying was stopped when the moisture content of the final sample reached 8.0% d.b.

Each experiment was repeated thrice. The moisture content of the walnut samples was calculated during the experiments based on the initial moisture content. The average moisture content and temperature of walnut kernels over three replicates were used to develop the drying curves. Immediately after the experiments, the dried walnut samples were vacuum packed into aluminum bags and stored in a refrigerator at −70°C for further quality analyses.

Water content determination and drying characteristics

The moisture content of walnuts was determined following the AOAC Official Method 925.40 with some modification. The ground walnut samples with less than 5 mm thickness were placed in the aluminum dishes and dried in a vacuum oven (DZX-6020B, Nanrong Laboratory Equipment Co., Ltd., Shanghai, China) at 100°C under 21.0 kPa for 7 h. Then the samples were placed in a desiccator with $CaSO_4$ and brought to room temperature before weighing. Each test was repeated in triplicate. The initial moisture content of shell, kernel, and whole walnuts was 24.5, 20.3, and 22.8% d.b., respectively.

Drying rate was defined as

$$DR = \frac{X_i - X_{i-1}}{\Delta t} \quad (1)$$

The change of moisture in walnuts during drying was expressed as moisture ratio (MR) defined as

$$MR = \frac{X_i - X_e}{X_o - X_e} \quad (2)$$

where MR is the dimensionless moisture ratio, t is time interval (min), X_i (kg/kg, d.b.) is the moisture content at any time i, X_0 is the initial moisture content (kg/kg, d.b.), and X_e is the equilibrium moisture content (kg/kg, d.b.), which was determined at the final stage of drying as an asymptotic value of the function fit to the experimental points.

Oil extraction and quality analyses

Both dried and fresh walnuts were manually cracked and shelled to remove the kernels. Grounded kernels (10 g) were placed in a Soxhlet apparatus containing 250 ml of petroleum ether (30–60°C) for 12 h. After leaching, the mixture was filtered, and the solvent was evaporated in a rotary evaporator under vacuum. The extracted oil was kept in a tube wrapped in aluminum foil at 4°C. All the quality tests were conducted within 2 h after extracting the oil.

Fatty acid composition

The analysis of composition and concentration of fatty acids in the walnut oil is useful for evaluating its stability, physical properties, and nutritional value. Fatty acid methyl esters (FAME)[23] were used to determine the fatty acid composition of the walnut oil. FAMEs were prepared by refluxing 50 µl of extracted oil with 5.0 ml of 1N NaOH solution in methanol. BF_3-methanol (5 ml) was added after 5 min boiling, followed by 5.0 ml heptane. After the two phases separated, the organic layer containing FAMEs was dried over a hydrous Na_2SO_4 and then transferred to a 1.0-ml tube for analysis by gas chromatography (7890A-5975C, Thermo Fisher Scientific, Waltham, USA) equipped with flame ionization detection and capillary DB-Wax column (30 m × 0.25 mm id × 0.25 µm film thickness, Agilent Technologies, J&W Scientific, Palo Alto, USA). The oven temperature was held from 180°C for 2 min, then programmed to increase to 240°C at 8°C/min, and finally held for 15 min. The injector and the detector temperatures were 230 and 250°C, respectively. The carrier gas was N2 at a flow rate of 1 ml/min, and the split radio was 80:1. Each fatty acid in the chromatogram was identified by comparing the retention times with certified standard mixes (Grain FAME Mix Supelco, Bellefonte, PA, USA; Catalog No: 47801) and quantified by peak area on the chromatogram using Chrom-Card data system version 2.3 software for Windows (Thermo Electron, Rodano, Italy).

Analysis of oil oxidative rancidity

The AV, PV, IV, and SV were calculated according to the AOAC standard methods[24] # Ca 5a-40, # Cd 8–53, # Cd 1–25, and #Cd 3b-76, respectively, using freshly extracted walnut oil.

Determination of total phenolic concentration and total antioxidant capacity

The extraction for antioxidant evaluations was prepared by placing approximately 3.0 g of macerated walnut kernels in a 50-ml centrifuge tube and homogenizing with 30 ml aqueous methanol (80%, v/v) for 1 h. The mixture was sonicated (40 kHz) for 20 min in the dark and then filtered in a Buchner funnel (90 mm i.d.) using #1 Whatman paper. The filtered solution was brought up to 50 ml with aqueous methanol and stored at −30°C until further analysis.

The TPC was measured using the Folin–Ciocalteu method[25] with some modifications. In brief, 0.2 ml of the extracted walnut solution in aqueous methanol was mixed with 0.2 ml of Folin–Ciocalteu reagent and 2.0 ml of deionized water. The mixture was kept at room temperature for 5 min. Then 2.0 ml of 0.18 mol/L Na_2CO_3 was added to the reaction mixture. A spectrophotometer (UV2000, Unico Instrument Co., Ltd, Shanghai, China) was used to record absorbance at 750 nm. The results are expressed as gallic acid equivalents (GAE) on a dry weight basis (mg GAE/g D.W.).

The TAC was evaluated by both the free radical (superoxide anion and 2,2-disphenyl-1-picrylhydrazyl, DPPH) scavenging capacity tests and reduced power (ferric reducing antioxidant power, FRAP) assays.

The superoxide anion scavenging capacity test was measured according to Noda et al.[26] DPPH free radical scavenging activity was measured according to the studies of Brandwilliams et al.[27] with minor modifications. Diluted sample of the walnut extract (0.1 ml) was added to a screw-cap tube containing 3.9 ml DPPH solution and then incubated in a dark water bath at 30°C for 30 min. The decrease in absorbance at 517 nm was recorded using a spectrophotometer (UV2000, Unico Instrument Co., Ltd, Shanghai, China). The results are expressed as trolox acid (6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid) equivalent antioxidant values (TEAC, µmol Trolox/g D.W.).

The reduced power FRAP of the walnut oil extraction was determined according to the method of Benzie et al.[28] with some modifications. Walnut extract (1.0 ml) was diluted 20 times, and then mixed with 2.5 ml of 0.2 mol/L phosphate buffer (PH 6.6) and 2.5 ml of 0.03 mol/L potassium ferricyanide (2.5 ml) was added, followed by 2.5 ml 0.6 mol/L trichloroacetic acid, and the mixture was centrifuged at 1,000×g for 10 min. The top layer of the solution (2.5 ml) was mixed with 2.5 ml of distilled water and 0.5 ml of 0.006 mol/L $FeCl_3$. The mixture was incubated at 37°C for 30 min and the absorbance at 700 nm was detected by spectrophotometer.

Oil storage experiment

For the walnut oil storage experiment, 50 ml of oil extracted from walnuts dried using AD, VD, and ARFD methods was poured into a 50-ml screw-cap plastic tube and subsequently the lid was closed. The tubes were stored in the dark at 35°C and 30% relative humidity in an incubator (BSC-150, Shanghai Boxun Industry & Commerce Co., Ltd, Shanghai, China) for 20 d. Subsamples were taken out every 10 d for quality analysis. Each storage test was conducted in triplicate. The accelerated storage experiments were conducted at 35°C with 30% relative humidity to simulate about 2 years of storage periods at 4°C or 2 months storage periods at room temperature (25°C). This was evaluated based on a Q_{10} value of 3.4 at 35°C.[29] Many accelerate shelf life experiments for almond,[30] walnut,[31], and rice[32] have been successfully conducted using the Q_{10} value. The following equation was used to calculate the Q_{10} value:

$$Q_{10}^{(T_1-T_2)/10} = \frac{\theta_S(T_1)}{\theta_S(T_2)} \quad (3)$$

where T_1 and T_2 are normal and accelerated temperatures (°C), respectively, and θ_s was storage time (day) of walnut oils.

Statistical analysis

The results are expressed as mean ±standard deviations over three replicates. Significant differences ($P < 0.05$) among means were estimated by the analysis of variance[33] and Turkey's significant difference test using the statistical software SPSS 16.0 version (SPSS Inc., Chicago, IL, USA) and Microsoft Excel variance procedure (Microsoft Office Excel, 2010).

Results and discussion

Effects of different drying methods on drying characteristics

The total drying time required for AD was the longest (300 min), followed by VD (185 min), and ARFD, which yielded the shortest time (138 min) (Figure 3a). Volumetric heating greatly increases the drying rate compared to surface heating with convection and internal heating with conduction.[22] Similar results were also reported for drying of lettuces stem slices using RF and hot air.[34]

Figure 3b shows the changes of kernel temperatures during the whole drying process. At the beginning, when moisture content was high, the temperature under ARFD condition increased rapidly with time, while the other two drying methods showed the slower temperature increasing rates due to the slow heating with convection and conduction. However, the temperatures of ARFD-treated samples later decreased when moisture content decreased. This may be due to the decreased absorption of RF energy due to decreased dielectric loss factor of relatively dried walnut samples. Moreover, hot air (50°C) lowered the temperature of the samples at this stage.

Change of drying rate is shown in Figure 3c, which shows that obvious differences in drying rate were found among the three drying methods. The removal of water by ARFD was very rapid initially when moisture content was high (MC 20.0% d.b.), and slowed when moisture content reached 9.8% d.b. because there was less available water to interact with RF energy in agrees with the results of previous studies.[35–37] VD showed the same trend of drying as ARFD, although the drying rate was less than that of ARFD. This was likely due to the lower heat conduction rate in VD, resulting in less thermal energy that is needed for moisture evaporation in the interior of walnut samples. However, a large vapor pressure gradient between the sample interior and the surface under vacuum may help to achieve much shorter drying times (about 40%) compared to AD. Similar results were also reported for drying of edamame[38] and wild cabbage[37] dried by VD compared to AD.

Effects of different drying methods on lipid oxidation of walnuts

During the drying period, the AV, PV, and SV all exhibited an increasing trend, while the IV decreased for all the drying methods and more rapidly in AD than in VD and ARFD. Qu et al.[39] also reported that walnuts dried using sun, direct oven, and intermittent oven showed an increasing trend in lipid rancidity.

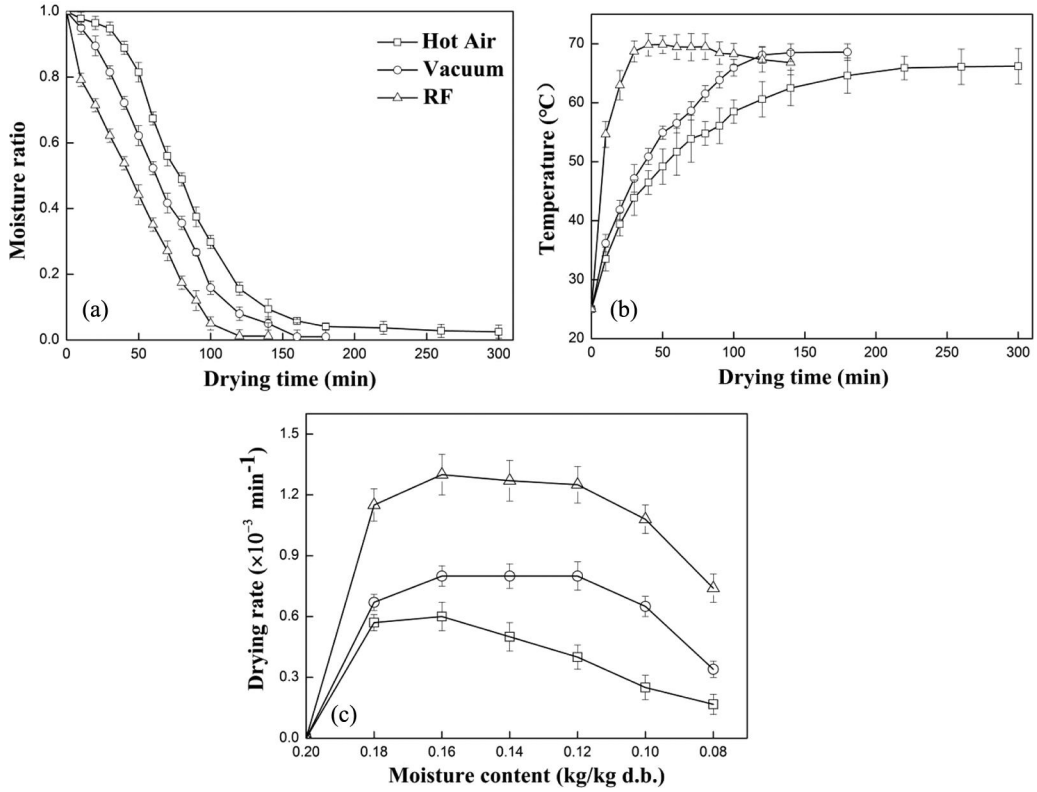

Figure 3. Changes in (a) moisture ratio, (b) temperatures, and (c) drying rate of walnut samples during the hot air, vacuum, and radio frequency drying processes.

The AV can be used to express the value of free fatty acids forming during the long storage periods due to oil hydrolytic rancidity. If the lipase activity was high, the acid values rose rapidly. The AV of oils extracted from AD-treated walnuts increased from 0.19 to 0.70 g/kg (Figure 4a), which was significantly higher than that of the other two drying methods ($P < 0.05$). However, there were no significant differences in AV between VD and ARFD ($P > 0.05$). During VD, the AV in walnut oils increased slightly from 0.19 to 0.43 g/kg mainly due to the reduced concentration of oxygen. Particularly, the AV in ARFD-treated samples increased rapidly from 0.19 to 0.44 g/kg until their moisture was reduced to 12% d. b., but the AV increased slowly thereafter and finally remained nearly constant value. This was likely due to the RF rapid heating. This result is in agreement with the study of Ling et al.[40] who reported that the AV in walnut paste increased from 0.29 to 0.35 g/kg after 10 min of RF heating. Overall, the walnut oils extracted from walnuts dried by ARFD and VD methods exhibited lower AV at the end of the drying period compared to AD.

The PV can be considered as a sign of oxidative rancidity, reflecting the value of hydroperoxide as fatty acid oxidation takes place. The mean PV of oil extracted from AD-treated walnut samples increased steadily from 0.31 meq/kg and reached the highest value of 0.56 meq/kg at the end of drying process (Figure 4b). However, the PVs of the oil extracted from walnuts dried by ARFD and VD methods were 0.38 and 0.33 meq/kg, respectively, lower than those dried by AD. Moreover, VD showed a relatively lower PV than ARFD. This was likely due to the low concentration of oxygen during the time that walnuts were exposed to an elevated temperature in the vacuum chamber. In addition, it has been reported that thermal treatments increase the PV of walnuts. For instance, Wang et al.[41] used 25 kW, 27 MHz RF system to control codling moth in in-shell walnuts and reported that the PV increased from 0.26 to 0.28 meq/kg due to 6 min of RF heating.

The SV is an important indicator of the liquidity and the hydrophilicity of the oil. The SV of oil extracted from AD-treated walnuts increased steadily until nut moisture content decreased to 14% d.b., then declined, and finally rose rapidly to 215.8 mg/g when the final sample moisture content of 8% d.b. was achieved. The trends of SV in walnuts treated by ARFD and VD were similar, both of which increased rapidly in the first 30 min, then decreased, and after about 120 min, SV increased again and finally reached

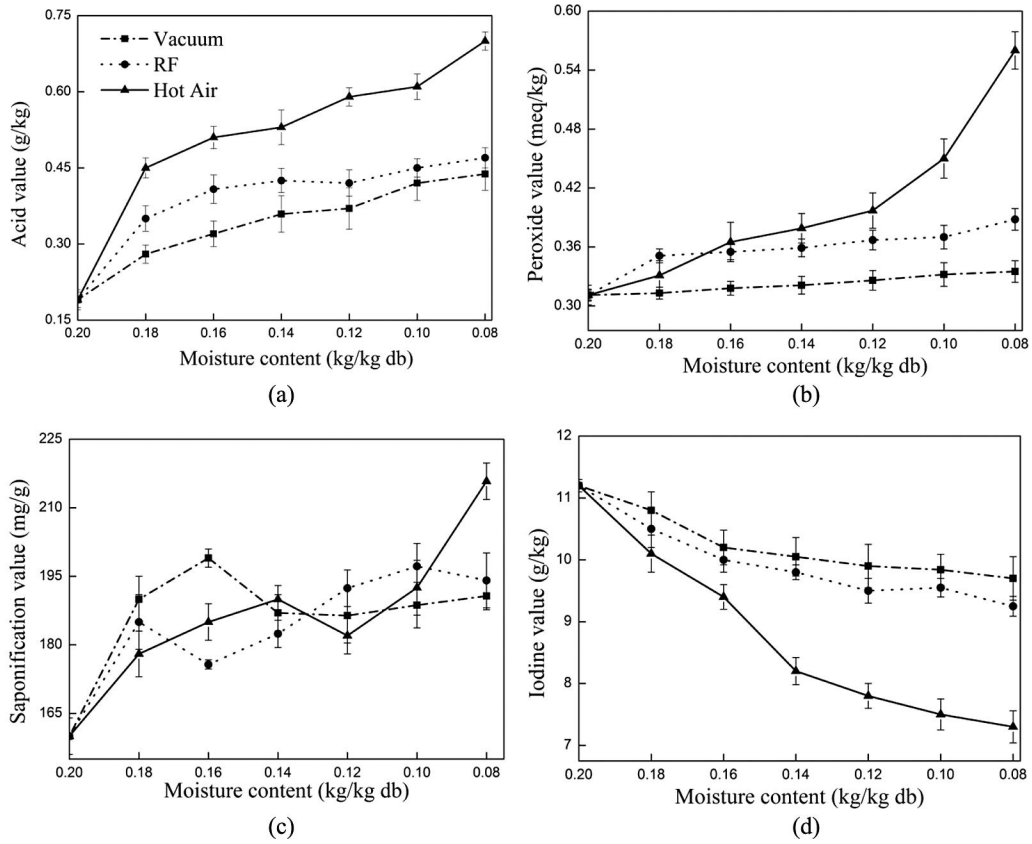

Figure 4. Changes in (a) acid value, (b) peroxide value, (c) saponification value, and (d) iodine value of oils extracted from walnut samples collected during three drying processes.

a peak at 194.1 and 190.7 mg/g at the end of drying, respectively (Figure 4c). This may be due to the changes and variability of free water and bound water during the entire drying process. These fluctuated results were also reported for walnuts dried by the oven.[39] Guldhe et al.[42] reported that the lipid obtained from freeze-dried, oven-dried, and sun-dried *Scenedesmus* showed higher SV values than that undried control samples. No significant differences in SV were found between ARFD- and VD-treated walnut samples ($P > 0.05$). In general, the AD-treated walnut samples had the highest values of SV among the three drying methods.

The IV is often used to determine the amount of unsaturation in fatty acids, as this unsaturation is in the form of double bonds (C=C), which react with iodine compounds. The higher IV, the more C=C bonds are present in the oil. The IVs of walnut samples dried by the three methods all exhibited a decreasing trend (Figure 4d). At the end of drying, the IVs of the walnuts were 9.7, 9.2, and 7.3 g/kg for VD, ARFD, and AD, respectively. However, there was no significant difference in IV between VD- and ARFD-treated walnut samples ($P > 0.05$).

In general, Figure 4 shows that qualities of walnut oils from VD and ARFD were similar although still had some significant differences ($P < 0.05$) in peroxide value, iodine value, etc., which was probably caused by the relatively stable fatty acids and antioxidant activity in the walnut oils. These stable qualities under different heating temperatures were also reported by Ling et al.[40] Moreover, drying time and temperatures have important effects on the product quality. The long drying times in AD at high temperature (70°C) during the long falling rate periods often led to undesirable thermal degradation. However, applying RF heating in hot air drying can significantly shorten process time and improve product quality. Vacuum drying, although took more time than ARFD, reduced lipid or other forms of oxidation due to the absence of air. Therefore, the qualities of walnut oils treated by RF and vacuum drying were better than those treated by hot air drying. VD and ARFD had the relatively same drying time and temperature over the entire drying process. In addition, samples under VD had better quality than those under ARFD due to the limitation of O_2 but not obvious. Because O_2 had little effect on the quality of walnut oils during the relatively short drying process.

Effects of different drying methods on fatty acid compositions

Data on the major fatty acids were used to analyze any change in oil composition as a function of drying method. The UFA content was more than 91% of the total, thereby making walnut oils highly susceptible to oxidation, which leads to rancidity and off-flavor. There was no significant difference ($P > 0.05$) in the fatty acid composition of oil from walnuts dried by the three drying methods (Table 1). Immediately after drying, linoleic acid (C18:2) was the most abundant polyunsaturated fatty acid (PUFA) at 59.21–61.36 g/100 g with a lower amount of linolenic acid (C18:3) at 7.63–10.49 g/100 g. Moreover, the major monounsaturated fatty acid (MUFA) found in walnut oil was oleic acid (C18:1) and ranged from 15.25 to 15.66 g/100 g. In comparison, the primary saturated fatty acids (SFA) present in walnut oils were palmitic acid (C16:0) and stearic acid (C18:0), which were present at lower concentrations (4.64–5.48 and 0.99–1.52 g/100 g, respectively). Overall, the total SFA, MUFA, and PUFA concentrations in each oil sample extracted from walnuts from three drying methods remained unchanged compared to control samples, suggesting that drying treatments had little effect on the composition and concentration of fatty acid in the walnut oils. This was likely due to the relative stability of fatty acids in the walnut oils. Similar results were also reported for walnut[43] and rice.[44]

Effects of different drying methods on total antioxidant capacity and total phenolic content

Among the common plant foodstuffs and especially nuts, walnuts rank at the top of the scale for their antioxidant capacities, as determined by both the ferric reduced power assay and free radical scavenging activity tests, since they exhibit high TPCs.[45]

The DPPH radical is composed of stable free radical molecules and is a useful reagent for analysis of the free radical scavenging ability. The DPPH free radical scavenging activity of walnut samples all increased initially and then decreased steadily (Figure 5a). At the end of drying process, DPPH free radical scavenging activity of walnuts treated by VD and ARFD remained unchanged compared to that of fresh samples, but that of samples from walnuts dried using AD decreased from 40 to 22%.

The superoxide anion radical scavenging assay is another important assay to evaluate the free radical scavenging ability of walnuts. All walnut samples showed an increasing trend in free radical scavenging ability until their moisture content decreased to about 16% d.b. (Figure 5b). The trends in superoxide anion radical scavenging activity of walnuts treated by VD and ARFD were similar, and both decreased slightly below the initial level. In general, AD samples showed the lowest superoxide anion radical scavenging activity among the three drying methods.

Ferric reducing antioxidant power assay is generally based on assessment of the transformation of Fe^{3+} to Fe^{2+}. Changes in FRAP in walnut samples treated by different drying methods are reflected by the absorbance at 700 nm (Figure 5c). The FRAP of oils from walnuts dried by AD increased slightly and reached a peak of 73% when their moisture content decreased to 12% d.b. and decreased sharply to 51% at the end of drying process, which was significantly lower than that of the other samples ($P < 0.05$). The trends of FRAP in VD- and ARFD-treated samples were similar, and both increased rapidly until their moisture content decreased to 14% d.b. and reached a maximum of 82 and 88%, respectively, then remained unchanged. In general, the FRAP of oils extracted from walnuts from VD and ARFD drying methods was enhanced, but that of AD-treated samples decreased slightly.

During drying, changes in TAC assessed by both reduced power assay and free radical scavenging activity followed a pattern similar with those of TPC (Figure 5d). This similarity could be explained by the fact that phenolic compounds contribute significantly to the antioxidant capacity, as observed by others.[46,47] Indeed, the TPCs of walnuts treated by VD, ARFD, and AD were 11.37, 11.51, and 9.66 mg/g, respectively. However, the percentage of loss in TAC was slightly

Table 1. Relative percentage of the main fatty acids (mean ± SD over 3 replicates) of oils extracted from the walnut kernels subjected to three drying methods.

Fatty acid	Fresh	AD	VD	ARFD
C16:0 (palmitic acid)	5.15 ± 0.24a*	4.64 ± 0.46a	5.48 ± 0.53a	5.05 ± 0.40a
C18:0 (stearic acid)	1.55 ± 0.23a	0.99 ± 0.21a	1.52 ± 0.25a	1.34 ± 0.18a
C18:1 (oleic acid)	16.07 ± 0.34a	15.25 ± 0.19a	15.66 ± 0.30a	15.50 ± 0.27a
C18:2 (linoleic acid)	60.58 ± 0.62a	59.21 ± 0.69a	61.36 ± 0.53a	60.41 ± 0.50a
C18:3 (linolenic acid)	10.84 ± 0.22a	7.63 ± 0.34b	10.49 ± 0.42a	10.12 ± 0.32a

*Values are means ± SD of three replicates. Means followed by different lowercase letters are significantly different at $P \leq 0.05$ among the AD, VD, and ARFD methods.

AD, hot air drying; VD, vacuum drying; ARFD, hot air-assisted radio frequency drying.

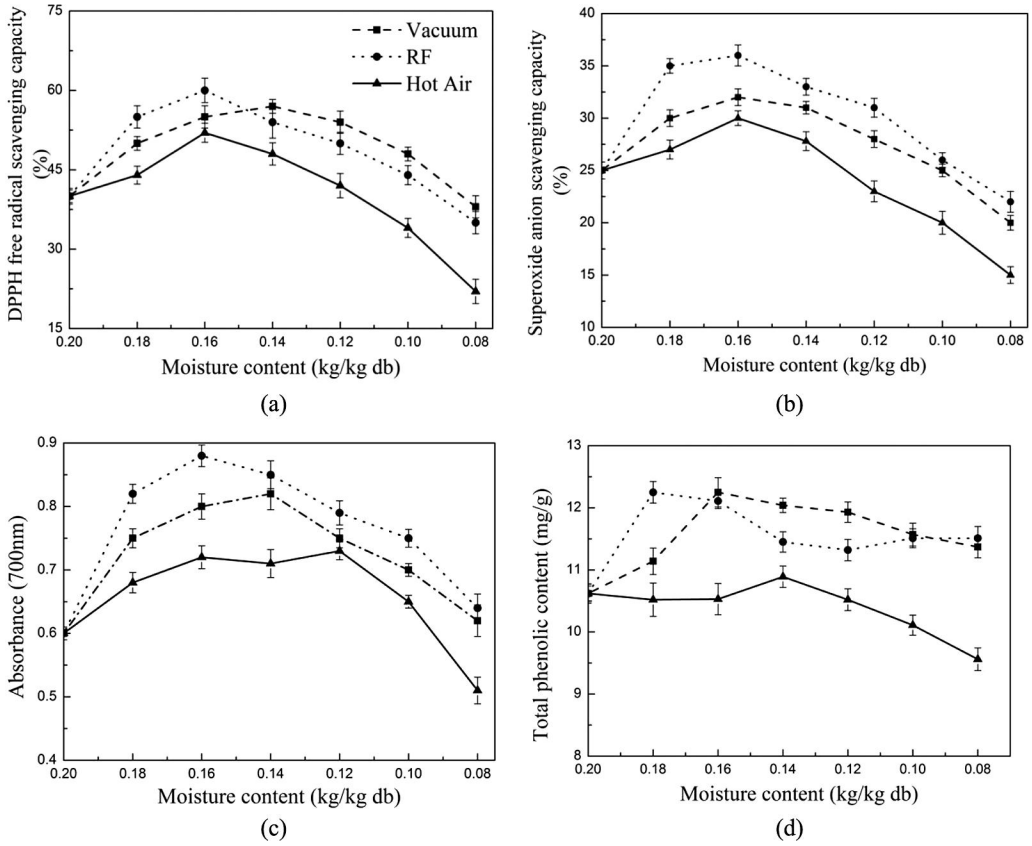

Figure 5. Changes in antioxidant capacities (a) DPPH radical scavenging activity, (b) superoxide anion scavenging activity, (c) ferric reducing power, and (d) total phenolic content of oils extracted from the walnut samples collected during three drying processes.

lower than that in TPC. This was likely due to the different quantitative changes in each type of phenolic compound during drying.[48] Moreover, flavonoids, such as rutin and quercetin, also show strong antioxidant activities.[49]

The similarity of the pattern of changes in TP, FRAP, DPPH, and superoxide anion could be explained by the fact that phenolics contributed significantly to TAC, as reported by others.[47,48] Moreover, in some cases, the partially oxidized polyphenols could exhibit higher antioxidant capacity than that of nonoxidized ones.[50] When the moisture content dropped down to around 0.16 kg/kg d.b., the polyphenols were partially oxidized, which led to the peaks of TAC. But with the drying time and temperature increased, the phenolic oxidation was responsible for a loss of antioxidant activity and the TAC decreased.

Quality analyses of walnut oils during storage

Lipid stability

Table 2 provides a detailed comparison of major lipid oxidation parameters (AV, PV, SV, and IV) in walnut oil extracted from walnuts dried using the AD, VD, and ARFD methods over the 20 d accelerated shelf-life storage at 35°C. There was no significant difference between oil from VD- and ARFD-treated walnuts for the four selected quality attributes ($P > 0.05$), except that the mean PV of ARFD-treated walnut sample was significantly higher ($P < 0.05$) than that of VD-treated walnut samples stored at 0 and 10 d and the IV of VD-treated samples was significantly higher than that of ARFD. Moreover, the AV, PV, and SV of the oils extracted from AD-treated walnuts were significantly higher ($P < 0.05$) than those from other two drying methods, and AD-treated oils showed the lowest IV over the entire period. Moreover, with increasing storage time, AV and SV increased rapidly for all treated samples after 20 d of accelerated storage, which is similar to results reported by Maskan et al.[51] in their storage tests for pistachio. In addition, the IV of dried walnut samples decreased with increasing storage time and reached a minimum value below 7.04 g/kg for all treated samples after 20 d of storage. In particular, the mean PV of samples exhibited an increasing trend after 10 d of storage and increased slightly or remained stable for 20 d of storage. These results are different from those observed by Labuckas et al.[52] and Vanhanen et al.[53] who showed that walnut oils remained stable or slightly fluctuated in a narrow range, which mainly

Table 2. Changes in lipid oxidation of oil extracted from walnuts subjected to three drying methods during accelerated storage at 35°C.

Lipid oxidation	Storage time (d) at 35°C	Drying method		
		AD	VD	ARFD
Acid value (mg/g)	0	0.69 ± 0.01aC*	0.42 ± 0.03cC	0.44 ± 0.02bcC
	10	2.95 ± 0.05aB	2.57 ± 0.04cB	2.67 ± 0.07bcB
	20	4.08 ± 0.04aA	3.70 ± 0.03cA	3.76 ± 0.05bcA
Peroxide value (meq/kg)	0	0.57 ± 0.02aB	0.33 ± 0.01cB	0.38 ± 0.01bC
	10	0.76 ± 0.04aA	0.51 ± 0.02cA	0.63 ± 0.02bA
	20	0.69 ± 0.05aA	0.55 ± 0.02cA	0.57 ± 0.01bcB
Saponification value (mg/g)	0	213.23 ± 1.43aC	189.03 ± 4.20cC	193.80 ± 3.60bcB
	10	237.92 ± 2.59aB	213.06 ± 5.78cB	219.54 ± 6.62bcA
	20	250.07 ± 1.85aA	224.60 ± 2.93bcA	223.80 ± 4.67cA
Iodine value (g/kg)	0	7.34 ± 0.15bA	9.74 ± 0.14aA	9.58 ± 0.08aA
	10	6.72 ± 0.13cB	8.54 ± 0.12aB	8.30 ± 0.05bB
	20	4.54 ± 0.09bC	6.98 ± 0.10aC	7.04 ± 0.13aC

*Values are means ± SD of three replicates. Means followed by different lowercase and uppercase letters are significantly different at $P \leq 0.05$ among the drying treatments and the storage time, respectively.
AD, hot air drying; VD, vacuum drying; ARFD, hot air-assisted radio frequency drying.

due to the protective effect of the antioxidant compounds. However, the results observed in this study are in general agreement with those reported by Wang et al.[31] and Mitcham et al.,[54] in which walnuts in the same storage condition developed very noticeable oxidative changes. Similarly, Martinez et al.[55] also reported that the walnut oils without drying showed lower oxidative damage than their treated counterparts during the entire storage period, suggesting that the spray drying process had diverse influences on some phenolic compounds. Above all, various results of oxidative stability were probably caused by different material cultivars, drying treatment, and storage conditions.

Fatty acid composition

Table 3 shows the fatty acid data in AD-, VD-, and ARFD-treated walnut oils over the 20 d of accelerated storage at 35°C. The concentration of palmitic acid (C16:0), stearic acid (C18:0), and oleic acid (C18:1) showed slight decreases during storage, while that of linolenic acid (C18:3) increased rapidly with storage time. Linoleic acid (C18:2) fluctuated in a narrow range for all treated samples during the storage period. However, there was no significant difference ($P > 0.05$) in fatty acid composition between VD- and ARFD-treated samples during storage except that the linoleic acid value and the linolenic value of the VD-treated walnuts were significantly higher than those of ARFD-treated samples stored at 20 d. These results were also observed by Ling et al.[56] and Zong et al.[57] indicating that RF and vacuum drying methods have little influence on the proximate fatty acid compositions of the nut oils. Meanwhile, oil from walnuts dried by the AD method showed significant differences ($P < 0.05$) in fatty acid concentrations from the samples treated by VD and ARFD.

Table 3. Relative percentages of the main fatty acids of oil extracted from walnut kernels subjected to three different dryings during accelerated storage at 35°C.

Fatty acid	Storage time (d) at 35°C	Drying method		
		AD	VD	ARFD
C16:0 (palmitic acid)	0	4.04 ± 0.46bA*	5.58 ± 0.33aA	5.05 ± 0.40aB
	10	3.69 ± 0.15bA	5.42 ± 0.20aA	5.54 ± 0.18aA
	20	3.75 ± 0.13bA	5.27 ± 0.12aA	5.35 ± 0.32aAB
C18:0 (stearic acid)	0	0.99 ± 0.11bA	1.52 ± 0.15aA	1.34 ± 0.18aA
	10	0.72 ± 0.05bB	1.39 ± 0.21aA	1.38 ± 0.14aA
	20	0.75 ± 0.16bAB	1.28 ± 0.17aA	1.14 ± 0.11aA
C18:1 (oleic acid)	0	15.25 ± 0.19bA	15.66 ± 0.13aA	15.50 ± 0.17abA
	10	13.39 ± 0.21bB	14.82 ± 0.16aB	14.78 ± 0.15aB
	20	12.47 ± 0.25bC	14.27 ± 0.14aC	14.14 ± 0.12aC
C18:2 (linoleic acid)	0	59.21 ± 0.69bA	61.36 ± 0.33aA	60.51 ± 0.71abA
	10	58.16 ± 0.55bA	60.84 ± 0.45aA	59.87 ± 0.51aA
	20	57.02 ± 0.43cB	61.15 ± 0.24aA	59.88 ± 0.31bA
C18:3 (linolenic acid)	0	7.63 ± 0.34bC	10.49 ± 0.42aC	10.12 ± 0.32aB
	10	8.55 ± 0.26bB	12.65 ± 0.25aB	12.74 ± 0.19aA
	20	10.71 ± 0.51cA	13.76 ± 0.35aA	12.57 ± 0.26bA

*Values are means ± SD of three replicates. Means followed by different lowercase and uppercase letters are significantly different at $P \leq 0.05$ among the drying treatments and the storage time, respectively.
AD, hot air drying; VD, vacuum drying; ARFD, hot air-assisted radio frequency drying.

Table 4. Changes in antioxidant activity and total phenolic content of oil extracted from walnuts subjected to three drying methods during accelerated storage at 35°C.

		Drying method		
Antioxidant activity	Storage time (d) at 35°C	AD	VD	ARFD
DPPH (%)	0	22.9 ± 2.2bA*	38.2 ± 1.8aA	35.7 ± 2.5aA
	10	21.2 ± 1.1bA	35.4 ± 1.7aA	36.1 ± 2.1aA
	20	18.6 ± 1.5bB	36.2 ± 2.1aA	34.2 ± 1.8aA
Superoxide anion (%)	0	17.5 ± 1.2bA	22.0 ± 1.8aA	23.5 ± 1.5aA
	10	16.5 ± 1.4bA	21.7 ± 0.7aA	22.1 ± 1.2aA
	20	16.3 ± 0.9bA	20.1 ± 1.5aA	21.2 ± 0.9aA
FRAP (%)	0	52.0 ± 2.1bBC	61.5 ± 1.1aC	62.4 ± 1.7aA
	10	61.4 ± 1.8cA	69.2 ± 0.9aA	65.4 ± 1.5bA
	20	48.2 ± 2.6bC	62.4 ± 1.5aBC	65.1 ± 2.2aA
TP (mg/g)	0	9.8 ± 0.8aA	11.4 ± 0.7aA	11.5 ± 0.9aA
	10	7.6 ± 1.4aAB	9.8 ± 0.9aAB	9.1 ± 1.1aBC
	20	6.3 ± 1.2bB	8.4 ± 0.5aB	8.2 ± 1.0abC

*Values are means ±SD of three replicates. Means followed by different lowercase and uppercase letters are significantly different at $P \leq 0.05$ among the drying treatments and the storage time, respectively.

AD, hot air drying; VD, vacuum drying; ARFD, hot air-assisted radio frequency drying; DPPH, DPPH radical scavenging activity; superoxide anion: superoxide anion scavenging activity; FRAP, ferric reducing power; TP, total phenolic content.

Antioxidant capacity and total phenolics

Table 4 shows the changes of TAC and TPC in oils from walnuts dried by three different methods over the 20 d of accelerated storage at 35°C. The trend of FRAP, DPPH, and superoxide anion scavenging capacities all followed a similar pattern during storage (Table 4). TPC decreased steadily with storage time. Indeed, the pattern with slight decreases in TPC was quite similar among the three drying methods, which indicated that oxidation occurred in phenolic compounds during storage. The results observed in this storage experiment are in general agreement with those of Christopoulos and Tsantili[48] who reported that the level of TPCs in walnut kernels decreased progressively with advanced storage time. In addition, there was no significant difference ($P > 0.05$) in TAC between VD- and ARFD-treated samples during storage except that the FRAP of VD-treated samples was significantly higher than that of ARFD-treated samples stored at 10 d. Except that the TP content of VD-treated samples was significantly higher than that of AD-treated samples stored at 20 d, there was no significant difference ($P > 0.05$) in total phenolic content among AD-, VD-, and ARFD-treated samples.

The TAC assessed by DPPH, superoxide anion or FRAP value remained unchanged during the entire storage period, but the TPC exhibited a decreasing trend by comparison. This discrepancy might be caused by that nonphenolic compounds contributing to antioxidant capacity, such as tocopherols. In addition, some studies indicated that the partially oxidized polyphenols may show higher antioxidant activity than nonoxidized polyphenols.[50] Furthermore, Zhang et al.[58] reported that the contribution of each phenolic compound, such as tocopherols and polyphenols, to antioxidant activity is different. A further experiment is needed to evaluate the effects of drying methods and storage time on tocopherols and polyphenols.

Conclusion

This study dealt with the effects of drying methods on drying characteristics and resulting in-shell walnut quality as measured by lipid oxidation, fatty acid composition, TPC, and TAC. The results showed that walnut samples from VD and ARFD exhibited lower lipid oxidation values than those of AD samples. Moreover, drying treatments using RF energy and vacuum had little effect on the TAC of walnut oils. In addition, walnuts treated by VD and ARFD had more UFA than those treated by AD. In particular, the ARFD method was associated with rapid heating and the shortest drying time (138 min) compared to VD (185 min) and AD (300 min). Therefore, ARFD provides a practical, effective, and rapid drying treatment protocol for in-shell walnuts while maintaining the walnut oil quality, especially the antioxidant capacity. A further study is desirable to analyze the drying characteristics of RF drying combined with vacuum and to study the effects of new treatment on the quality of walnuts.

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