LWT - Food Science and Technology 59 (2014) 786–793

Contents lists available at ScienceDirect

LWT - Food Science and Technology

journal homepage: www.elsevier.com/locate/lwt

Thermal treatment and storage condition effects on walnut paste quality associated with enzyme inactivation

Bo Ling [a], Lixia Hou [a], Rui Li [a], Shaojin Wang [a, b, *]

[a] Northwest A&F University, College of Mechanical and Electronic Engineering, Yangling, Shaanxi 712100, China
[b] Department of Biological Systems Engineering, Washington State University, 213 L.J. Smith Hall, Pullman, WA 99164-6120, USA

ARTICLE INFO

Article history:
Received 17 February 2014
Received in revised form
19 June 2014
Accepted 7 July 2014
Available online 16 July 2014

Keywords:
Thermal treatment
Walnuts paste
Oxidative rancidity
Hydrolytic rancidity
Storage

ABSTRACT

In this study, walnut paste was heated at 60, 75, and 90 °C with three selected times for enzyme inactivation, and its quality stability was determined under accelerated storage conditions by evaluating peroxide value (PV), conjugated diene value (CDV), fatty acid value (FFA) and fatty acid composition. The results showed that the mean PV, CDV and FFA values of the heated paste samples increased with increasing treatment temperature and time. The PV and CDV were reduced or stable, while the FFA values increased significantly during storage period. However, no significant quality difference was observed between controls and heated walnut paste samples with vacuum package after accelerated storage for 20 days. Compared with unheated walnut kernels and cold-pressed oils in vacuum or normal package under same storage conditions, the mean PV of the unheated paste were the lowest, while its mean FFA values were the highest. FFA values of the paste were still lower than the acceptable range (FFA < 0.6%) used by industry for good walnut quality, suggesting that vacuum or normal packages can be used to keep quality of walnut paste for 20 days at 35 °C simulating 2 years of storage at 4 °C or 2 months at 25 °C.

1. Introduction

Walnut (*Juglans regia* L.) is a tree nut with high economic values to the food industry. The world production of in-shell walnuts was around 3.282 Mt in 2012 and China is the leading world producer with about 50% of the total world production (FAOSTAT, 2012). The edible kernel represents 40–60 g/100 g of the nut weight, which is commonly used as functional ingredients in food products, such as candy, chocolate, breads, cake, etc (Bakkalbasi, Yilmaz, Javidipour, & Artik, 2012). With the increasing demand for ready-to-eat processed foods with extended shelf life, pleasant taste, easy portability and high nutritional quality, walnut products in various forms such as oils, paste and defatted flour have been developed continuously (Ayo, Carballo, Solas, & Jiménez-Colmenero, 2005; Cofrades, Serrano, Ayo, Carballo, & Jiménez-Colmenero, 2008; Martinez, Barrionuevo, Nepote, Grosso, & Maestri, 2011; Martinez & Maestri, 2008).

Walnut kernel is a nutrient-rich food mainly owing to its high-biological-value proteins (low lysine/arginine ratio), high levels of oil (60 g/100 g in average mainly polyunsaturated fatty acids, PUFA) and antioxidants (phytosterols and polyphenols) (Shimoda et al., 2008; Venkatachalam & Sathe, 2006; Zwarts, Savage, & Mcneil, 1999). Although fatty acid compositions in walnuts are favorable from a nutritional point of view, higher contents of PUFA may result in poorer quality stability and shorter shelf life of walnut products.

Watkins (2005) reported that the quality stability of walnut kernels during storage is limited mainly by rancidity development because of oxidation and lipolysis. Environmental factors such as oxygen, storage temperature, relative humidity, and light are major factors to accelerate lipid oxidation (Crowe & White, 2003; Mate, Saltveit, & Krochta, 1996; Mexis, Badeka, Riganakos, Karakostas, & Kontominas, 2009). Lipolysis caused by enzymes such as lipase (LPS), peroxidase (POD) and lipoxygenase (LOX) is another important factor that could play an essential role in rancidity of walnut kernels (Piccirillo, Fasano, Mita, De Paolis, & Santino, 2006; Piffaut & Metche, 1991; Yeşiloğlu & Demirkan, 2010; Zacheo, Cappello, Gallo, Santino, & Cappello, 2000).

Various storage procedures have been developed for maintaining the post-harvest quality of walnut kernels. Lopez, Pique, Romero, and Aleta (1995) found that the whole walnut quality

* Corresponding author. Northwest A&F University, College of Mechanical and Electronic Engineering, Yangling, Shaanxi 712100, China. Tel.: +86 29 87092319; fax: +86 29 87091737.
E-mail addresses: shaojinwang@nwsuaf.edu.cn, shaojin_wang@wsu.edu (S. Wang).

http://dx.doi.org/10.1016/j.lwt.2014.07.005
0023-6438/

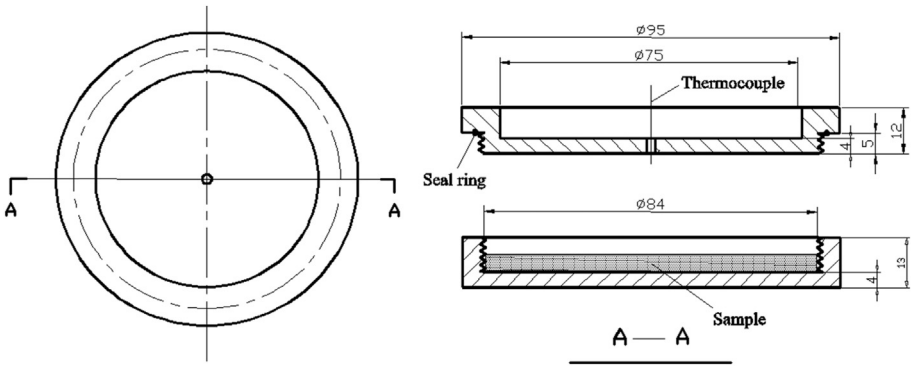

Fig. 1. Schematic diagram of a hermetically sealed test cell (All dimensions are in mm).

was acceptable after 12 months storage at 10 °C and 60% relative humidity. Mexis et al. (2009) reported that key factors influenced the quality of shelled walnuts during storage in following order: temperature > degree of O_2 barrier > lighting conditions. Mate et al. (1996) reported that lipid oxidation was inhibited by using a packaging material with low oxygen permeability or by storing the walnuts in controlled atmospheres with low oxygen content.

Thermal processing as a pre-storage treatment applied for inhibiting enzymes activity has been studied for several nut products. Mitchell and Malphrus (1977) reported that the LOX was not found in whole shelled peanuts after steam heating at 100 °C for 2 min, and very little POD activity could be detected for 30 min. LPS in hazelnuts was reported to be inactivated by saturated steam treatment for 20 min (Laskawy, Senser, & Grosch, 1983). Senter, Forbus, Nelson, Wilson, and Horvat (1984) studied the pecan kernels exposed to dielectric or steam heating and reported that heat treatments in the range of 90–100 °C would be optimal for stabilization of pecan kernel quality during storage, probably because of partial inactivation of enzymes. Zacheo et al. (2000) reported that the LOX activity of the crude extracts of almonds was lost after 10 min exposure to 80 °C. Cam & Kilic (2009) reported that the blanching treatment at 120 °C for 15 min could improve the oxidative stability of hazelnut meal. Buranasompob, Tang, Mao, and Swanson (2003) found that hot air heating of walnut and almond kernels at 60 °C for up to 10 min did not increase rancidity, which might be due to possible inactivation of the LOX by thermal treatments. Although substantial research has been performed about effects of thermal treatment on enzyme inactivation in different nut products, studies related to thermal inhibition of enzyme activity together with storage stability of walnut paste are limited.

The objectives of this study were: (1) to investigate the lipid stability of walnut paste as affected by heat treatment at three selected temperatures and holding times, (2) to evaluate the effects of different heating treatments on the storage stability of walnut paste under accelerated storage conditions, and (3) to compare the lipid stability of walnut kernels, paste, and cold-pressed oils with different package during accelerated storage.

2. Materials and methods

2.1. Materials

Walnuts (*Juglans regia* L.) with green husks were harvested from a commercial orchard in Yangling, Shaanxi, China, in September, 2013. To ensure uniform maturity and quality of samples, the walnuts were prescreened by sample size and then transported to the laboratory within 2 h. The green husks were peeled off within 1 d and the in-shell nuts were washed with tap water. After cleaning, the nuts were dried under direct sunlight about 10 h per day and ambient air conditions for three days to obtain kernel moisture content of about 4.5 g/100 g in wet basis. The nuts in-shell with uniform size and without mechanical damage and pest infestations were randomly selected and placed into polyethylene bags and stored at 2–4 °C and 65–70% relative humidity. Dried walnuts were shelled manually and whole kernels were stored at the same condition before conducting heat treatments.

2.2. Thermal treatments

To minimize the influence of slow heat transfer on sample quality during the heating up time, walnut paste was heated in a custom-designed test cell, which is illustrated in Fig. 1. The cells had a net inner space of 84 mm in diameter and 4 mm in depth. This test cell made by two pieces of aluminum alloy lids with a rubber o-ring on the top lid, could provide adequate strength and hermetic seal when a moist sample was heated at high temperatures, and ease in loading and unloading liquid and solid samples. The temperature range of 60–90 °C was selected based on enzyme inactivation (Mitchell & Malphrus, 1977; Senter et al., 1984). A pre-calibrated thin type-T thermocouple sensor was inserted through a rubber gland in the top lid to record the temperature-time history of sample core using a data logger (CR-1000, Campbell Scientific. Inc., Logan, Utah, USA). Because of the high aspect ratios of surface area to volume and the high thermal conductivity (180 Wm^{-1} K^{-1}) of aluminum alloy, the come-up time (CUT: the time needed for the geometric center of a sample temperature to reach 0.5 °C less than the set point) in this test cell was small, resulting in a close-to-ideal isothermal condition. Similar test cells have been successfully used to study the quality changes of salmon, blue mussel and purple-fleshed potato during thermal processing (Kong, Tang, Rasco, & Crapo, 2007; Nayak, Berrios, Powers, & Tang, 2011; Ovissipour, Rasco, Tang, & Sablani, 2013).

Walnut kernels were ground in a blender (JYL-D022, Joyoung, Jinan, China) to obtain paste samples. About 24 g paste was filled into the cells, and then the cells were sealed and heated in a digitally controlled constant-temperature water bath (SC-15, Scientz Biotechnology Co., Ltd., Ningbo, China) at the specified temperatures. Table 1 shows the temperature and time conditions employed,

Table 1
Treatment temperature and holding time for walnut paste.

Temperature (°C)	Heating time (min)		
60	10	20	30
75	10	15	20
90	5	10	15

which were selected based on the requirements for inhibiting enzyme activity in nuts (Laskawy et al., 1983; Mcglamery & Hood, 1951; Mitchell & Malphrus, 1977; Zacheo et al., 2000). When reaching the set-point temperature, the holding time was counted. After heating, the cells were immersed immediately into an ice water to cool down the samples till the central sample temperature was below 25 °C. After cooling, the paste was removed from the cell for oil quality analysis or storage treatment.

2.3. Storage experiment

Heat treated walnut paste and unheated walnut paste or kernels (140 g each) were packaged in two different methods: a) vacuum package under 93.3 kPa in polyamide/polyethylene laminate pouches having 90 μm total thickness (90 M PA/PE); b) normal package in a glass beaker (500 mL), which was loosely covered with filter paper to allow air to pass through. For walnut oil storage, 50 mL of cold-pressed oils were poured into 50 mL screw-cap test tubes and then the lid was closed tightly. All the samples were stored in the dark at 35 °C and 30% relative humidity (RH) for 20 d in an incubator (BSC-150, Shanghai Boxun Industry & Commerce Co., Ltd, Shanghai, China) and samples were taken at 5-day intervals for quality analysis. All the experiments were conducted in triplicate. The storage conditions at 35 °C for 20 d were chosen to accelerate storage tests, and simulate approximately 2 years storage periods at 4 °C or 2 months storage periods at ambient temperature (25 °C) based on a Q_{10} value of 3.4 at 35 °C (Taoukis, Labuza, & Saguy, 1997). The Q_{10} concept has been successfully used to design the accelerate shelf life tests for walnuts, almonds and powdered guavira pulp (Breda, Sanjinez-Argandoña, & Correia, 2012; Gao, Tang, Wang, Powers, & Wang, 2010; Wang et al., 2006) and calculated as:

$$Q_{10}^{(T_2-T_1)/10} = \frac{\theta_s(T_1)}{\theta_s(T_2)} \quad (1)$$

where T_1 and T_2 are normal and accelerated storage temperatures (°C), respectively, and θ_s stands for the storage time (d) of walnuts.

2.4. Oil extraction and quality analyses

The oil was extracted from the walnut paste or kernels using a hydraulic oil press (Lefen, Dezhou, China) without additional heat treatment. About 50 g of oil were obtained from approximately 140 g of walnut paste. The extracted oil was centrifuged at 2862 × g for 1 min. The supernatant oil was then transferred into amber glass containers at 4 °C. All the quality analyses were performed within 3 h after pressing. Quality parameters were peroxide values (PV), and conjugated diene values (CDV) for assessing oxidative rancidity, or fatty acid values (FFA) and fatty acid composition for analyzing hydrolytic rancidity.

2.4.1. Peroxide value (PV)

Peroxide value was determined by official method Cd 8-53 and recommended practices of the American Oil Chemists Society (AOAC, 1997). This method determines iodine liberated from potassium iodide by the peroxides present in the oil:

$$\mathrm{PV(meq/kg)} = \frac{(S-B)\times N\times 1000}{m} \quad (2)$$

where: B is the volume (mL) of titrant for blank, S is the volume (mL) of titrant for sample, N is the normality of $Na_2S_2O_3$ solution (0.1mol equi/L), and m is the weight (g) of oil sample.

2.4.2. Conjugated diene values (CDV)

The CDV were determined according to the method reported by Chandrasekara and Shahidi (2011). In brief, a specified amount of oil (~0.2 g) was weighed into a 25 mL volumetric flask, and made up to the mark with 2,2,4-trimethylpentane. The solution was thoroughly mixed before reading its absorbance at 233 nm using a UV–Vis spectrophotometer (Model UV2000, UNICO Co., Ltd., Shanghai, China). Pure 2,2,4-trimethylpentane was used as a reference. CDV were calculated using the following equation:

$$\mathrm{CDV} = \frac{A_{233}}{C\times L} \quad (3)$$

where: A_{233} is the absorbance of solution at 233 nm, C is the concentration of oil in g per 100 mL, and L is the length of the cuvette in cm.

2.4.3. Fatty acid values (FFA)

FFA was analyzed by official method Ab 5-49 and recommended practices of the American Oil Chemists Society (AOAC, 1997). FFA was a quantitative determination of fatty acids in oil by titration against a standardized alkali solution (NaOH) as follows:

$$\mathrm{FFA\ (Oleic\ acid\ g/100g)} = \frac{V\times N_1\times 28.2}{m} \quad (4)$$

where: V is the titration of sample (mL), and N_1 is the normality of NaOH solution.

2.4.4. Free fatty acid composition

The fatty acid composition of an oil can be used as an indicator of its stability, physical properties, and nutritional value (Crews et al., 2005), which could be determined by forming fatty acid methyl esters (FAME) (Arena, Campisi, Fallico, & Maccarone, 2007). FAMEs were prepared by refluxing 0.05 g of oil samples with 5 mL of 1 N sodium hydroxide solution in methanol. Five minutes after boiling, 5 mL of BF_3-methanol were added, and followed by 5 mL of heptanes after another 5 min. After cooling the mixture, 5 mL of saturated NaCl solution were added and the organic layer with the FAMEs was recovered and dried over a hydrous Na_2SO_4 and then analyzed by gas chromatograph (GC). FAME were separated on a capillary DB-Wax column (30 m × 0.25 mm id × 0.25 μm film thickness, Agilent Technologies, J&W Scientific, USA) by using a GC (Thermo Fisher Scientific, USA) equipped with flame ionization detection (FID). The oven temperature was programmed from 180 °C for 2 min and then to 240 °C at 8 °C/min. The temperatures of injector and detector were 230 and 250 °C, respectively. Nitrogen was used as the carrier gas at a flow rate of 1 mL/min, with an injection volume of 1 μL using a split ratio of 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; Catlog No: 47801) and quantified by the peak areas on the chromatogram using Chrom-Card data system version 2.3 software for Windows (Thermo Electron, Rodano, Italy). The results were expressed as g of FA per 100 g of total FAs. Polyunsaturated fatty acid (PUFA) contents were calculated as sums of individual fatty acids.

2.5. Statistical analysis

Results were expressed as means ± standard deviations (SD) over three independent experiments. Differences were estimated by the analysis of variance (ANOVA) followed by Tukey's test and considered significantly at $p \leq 0.05$. All statistical analyses were performed using the statistical software SPSS 16.0 version (SPSS Inc., Chicago, IL, USA).

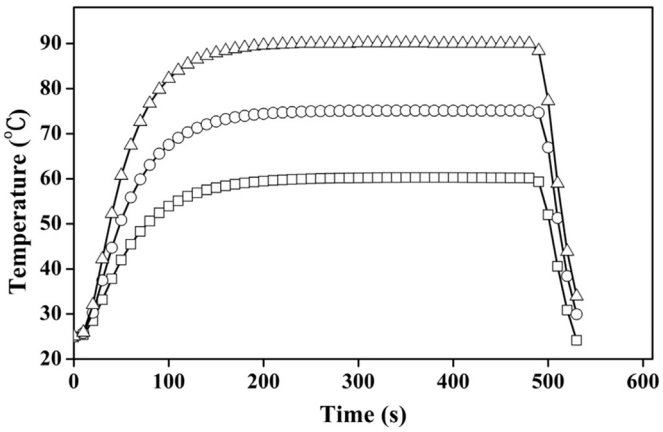

Fig. 2. Typical temperature-time history for walnut kernel paste samples in the test cell center (□60 °C, ○75 °C, △90 °C).

3. Results and discussion

3.1. Heat treatments

Fig. 2 shows temperature-time histories for the central samples in the custom-designed test cell at three selected temperatures. Come-up-times averaged approximately 200 s for all the test temperatures, after which the samples experienced close to ideal isothermal exposure. The sample temperature during holding time was relatively constant and the cooling process took less than 60 s for sample core temperatures to drop to less than 25 °C. Kong et al. (2007) used similar test cell (⌀: 30 mm × H: 6 mm) to investigate the salmon quality changes in high temperature range of 100 ~ 131 °C, the CUT was on the order of 2.5–3 min. Ovissipour et al. (2013) also used similar test cells (⌀: 50 mm × H: 8 mm) to evaluate the quality changes in whole blue mussel over the temperature range of 65 ~ 90 °C, the CUT was on the order of 6.8–4.7 min. Many factors have influence on CUT and cooling time, in which the sizes of test cell, set temperature, cooling medium and thermal properties of food products are the major ones. The short CUT using the test cell may ensure reliable and repeatable quality analyses.

3.2. Chemical analyses for oxidative rancidity of walnut paste

The initial mean PV was 0.40 meq/kg for unheated walnut paste without storage, which was significantly lower ($p < 0.05$) than that (0.51–0.72 meq/kg) for heated walnut paste stored at 0 day (Table 2). The maximum PV after heat treatments was still lower than the limited value (1.0 meq/kg) for good walnut quality recommended by walnut industry in California, USA (Wang, Monzon, Johnson, Mitcham, & Tang, 2007). Generally, the initial mean PV increased with treatment temperature and time except for some PV variations caused by heterogeneous nature of the samples. The PV of heated walnut paste decreased significantly with increasing storage time and reached a minimum value below 0.27 for all treated samples after 20 d of accelerated storage. The results described above are completely different from those reported by Mitcham et al. (2004) and Wang et al. (2006, 2007) in which in-shell walnuts in same storage conditions could develop pronounced oxidative changes. However, the results observed in this experiment are in general agreement with those reported by Vanhanen and Savage (2006) who showed that the oil in the walnut flour slightly decreased or remained stable for up to six-months storage, probably because the flour contained high levels of vitamin E, a natural antioxidant. Similarly, Labuckas, Maestri, and Lamarque (2011) found that defatted walnut flour with aluminum-coated packages can be stored at 25 °C for 8 months period without any major changes of PV. Under a flour matrix, the remaining oils could be protected by phenolic compounds, mostly present in the pellicle, which persist in the flour after processing.

The initial mean CDV of oils extracted from heated walnut paste ranged from 0.41 to 0.48, which was significantly higher ($p < 0.05$) than that (0.33) for unheated samples. This trend was similar to those obtained for PV. However, the initial mean CDV of oils extracted from walnut paste heated at 90 °C were slightly higher than those at 60 and 75 °C, which exhibited insignificant difference ($p > 0.05$) in walnut paste (Table 3). The CDV of control samples increased slightly but those for heated samples decreased generally with increasing storage time. There was no significant ($p > 0.05$) effect of heating on CDV among different paste samples after 20 d of accelerated storage. Labuckas et al. (2011) reported that the CDV from defatted walnut flour with aluminum-coated packages did not vary significantly along storage period even after stored at 25 °C for 8 months. In contrast, Cai, Cao, Aisikaer, and Ying (2013) and Chandrasekara and Shahidi (2011) reported that the CDV of pine nut oils and cashew nut oils in glass vials stored at 60 °C increased significantly with increasing storage time. Various results of oxidative stability were probably caused by different material properties, storage temperatures, and packaging methods.

3.3. Chemical analyses for hydrolytic rancidity of walnut paste

FFA values are often used as an indicator for lipid hydrolysis by lipase, and the hydrolysis may be promoted by reaction of oil with moisture. The initial mean FFA value of oils extracted from unheated walnut paste was 0.12 g/100 g, but that from heated walnut paste ranged from 0.11 to 0.19 g/100 g. Generally, the mean FFA

Table 2
Peroxide value (PV, meq/kg oil) changes (mean values ± standard deviation over three replicates) of walnut paste packed under vacuum with different heating condition during accelerated storage.

Treatment	Storage time (d) at 35 °C				
	0	5	10	15	20
Control	0.40 ± 0.03^{dBC}	0.48 ± 0.03^{abA}	0.42 ± 0.02^{aAB}	0.34 ± 0.01^{abC}	0.22 ± 0.02^{aD}
60°C/10 min	0.51 ± 0.03^{cA}	0.43 ± 0.03^{abcB}	0.35 ± 0.03^{abC}	0.33 ± 0.01^{abCD}	0.26 ± 0.01^{aD}
60°C/20 min	0.60 ± 0.02^{bA}	0.37 ± 0.02^{cB}	0.33 ± 0.02^{abB}	0.34 ± 0.02^{abB}	0.27 ± 0.03^{aC}
60°C/30 min	0.72 ± 0.04^{aA}	0.53 ± 0.08^{aB}	0.34 ± 0.03^{abDE}	0.36 ± 0.04^{abCD}	0.24 ± 0.00^{aE}
75°C/10 min	0.60 ± 0.03^{bcA}	0.50 ± 0.02^{abB}	0.39 ± 0.02^{aC}	0.29 ± 0.04^{bCD}	0.23 ± 0.02^{aD}
75°C/15 min	0.63 ± 0.01^{bA}	0.41 ± 0.02^{bcB}	0.35 ± 0.03^{abBC}	0.32 ± 0.02^{abC}	0.25 ± 0.03^{aD}
75°C/20 min	0.63 ± 0.04^{bA}	0.42 ± 0.02^{abcB}	0.28 ± 0.04^{bC}	0.30 ± 0.03^{bC}	0.21 ± 0.02^{aC}
90°C/5 min	0.62 ± 0.03^{bA}	0.44 ± 0.04^{abcB}	0.35 ± 0.02^{abC}	0.37 ± 0.02^{aBC}	0.23 ± 0.03^{aD}
90°C/10 min	0.67 ± 0.01^{abA}	0.52 ± 0.06^{aB}	0.36 ± 0.03^{abC}	0.38 ± 0.03^{aC}	0.21 ± 0.01^{aD}
90°C/15 min	0.67 ± 0.04^{abA}	0.46 ± 0.02^{abcB}	0.40 ± 0.04^{aBC}	0.37 ± 0.02^{aC}	0.26 ± 0.01^{aD}

Mean values are not significantly different ($p > 0.05$) for the same capital letters within a row among the storage time, and for the same lower case letters within a column among the heating treatments.

Table 3
Conjugated diene (CD) value changes (mean values ± standard deviation over three replicates) of walnut paste packed under vacuum with different heating during accelerated storage.

Treatment	Storage time (*d*) at 35 °C				
	0	5	10	15	20
Control	0.33 ± 0.02^{eC}	0.39 ± 0.03^{dAB}	0.42 ± 0.02^{abA}	0.37 ± 0.01^{bcBC}	0.37 ± 0.03^{abBC}
60°C/10 min	0.41 ± 0.01^{cdBC}	0.47 ± 0.02^{aA}	0.43 ± 0.03^{aAB}	0.38 ± 0.01^{bC}	0.37 ± 0.04^{abC}
60°C/20 min	0.45 ± 0.02^{abcdA}	0.42 ± 0.02^{bcdA}	0.37 ± 0.03^{bcB}	0.38 ± 0.01^{B}	0.39 ± 0.02^{abB}
60°C/30 min	0.41 ± 0.03^{dAB}	0.44 ± 0.02^{abcA}	0.40 ± 0.01^{abcAB}	0.39 ± 0.02^{bB}	0.35 ± 0.03^{bC}
75°C/10 min	0.42 ± 0.01^{cdA}	0.42 ± 0.03^{bcdA}	0.37 ± 0.03^{cB}	0.43 ± 0.01^{aA}	0.38 ± 0.01^{abB}
75°C/15 min	0.45 ± 0.02^{abcdA}	0.45 ± 0.02^{abA}	0.38 ± 0.01^{bcB}	0.44 ± 0.01^{aA}	0.40 ± 0.02^{aB}
75°C/20 min	0.44 ± 0.03^{bcdA}	0.40 ± 0.02^{cdB}	0.41 ± 0.02^{abAB}	0.42 ± 0.01^{aAB}	0.39 ± 0.03^{abB}
90°C/5 min	0.46 ± 0.02^{abA}	0.40 ± 0.02^{cdB}	0.38 ± 0.01^{bcBC}	0.37 ± 0.02^{bcC}	0.36 ± 0.02^{abC}
90°C/10 min	0.48 ± 0.02^{aA}	0.41 ± 0.02^{bcdB}	0.36 ± 0.01^{cC}	0.35 ± 0.01^{cC}	0.35 ± 0.01^{bC}
90°C/15 min	0.46 ± 0.02^{abcA}	0.38 ± 0.03^{dB}	0.41 ± 0.01^{abB}	0.38 ± 0.01^{bB}	0.39 ± 0.02^{abB}

Mean values are not significantly different ($p > 0.05$) for the same capital letters within a row among the storage time, and for the same lower case letters within a column among the heating treatments.

Table 4
FFA (Oleic g/100 g) changes (mean values ± standard deviation over three replicates) of walnut paste with different heating during accelerated storage.

Treatment	Storage time (*d*) at 35 °C				
	0	5	10	15	20
Control	0.12 ± 0.02^{bD}	0.27 ± 0.02^{cC}	0.41 ± 0.01^{abB}	0.46 ± 0.03^{abB}	0.52 ± 0.01^{aA}
60°C/10 min	0.11 ± 0.03^{bD}	0.32 ± 0.02^{aC}	0.43 ± 0.03^{aB}	0.46 ± 0.01^{abcAB}	0.51 ± 0.01^{aA}
60°C/20 min	0.11 ± 0.01^{bE}	0.29 ± 0.00^{abcD}	0.37 ± 0.02^{bcC}	0.44 ± 0.01^{abcB}	$0.52 \pm 0.02_{aA}$
60°C/30 min	0.12 ± 0.01^{bE}	0.26 ± 0.00^{cD}	0.35 ± 0.01^{cdC}	0.45 ± 0.00^{abcB}	0.52 ± 0.01^{aA}
75°C/10 min	0.13 ± 0.01^{bC}	0.29 ± 0.01^{abcB}	0.31 ± 0.01^{dB}	0.40 ± 0.02^{cA}	0.49 ± 0.03^{aA}
75°C/15 min	0.15 ± 0.02^{abE}	0.27 ± 0.01^{cD}	0.34 ± 0.01^{cdC}	0.42 ± 0.01^{abcB}	0.51 ± 0.02^{aA}
75°C/20 min	0.16 ± 0.02^{abE}	0.28 ± 0.01^{bcD}	0.34 ± 0.01^{cdC}	0.44 ± 0.05^{abcB}	0.52 ± 0.01^{aA}
90°C/5 min	0.12 ± 0.02^{bD}	0.32 ± 0.01^{aC}	0.35 ± 0.02^{cdC}	0.42 ± 0.01^{bcB}	0.50 ± 0.03^{aA}
90°C/10 min	0.15 ± 0.02^{abE}	0.29 ± 0.00^{abcD}	0.37 ± 0.01^{bcC}	0.47 ± 0.01^{aB}	0.51 ± 0.01^{aA}
90°C/15 min	0.19 ± 0.01^{aD}	0.32 ± 0.02^{abC}	0.42 ± 0.01^{abB}	0.46 ± 0.02^{abA}	0.50 ± 0.01^{aA}

Mean values are not significantly different ($p > 0.05$) for the same capital letters within a row among the storage time, and for the same lower case letters within a column among the heating treatments.

value increased with increasing treatment temperature and time at each storage day. The mean FFA values for both control and heated walnut paste increased with storage time. No significant differences ($p > 0.05$) were observed among initial treatment samples, except for heated samples at 90 °C for 15 min (Table 4). However, no significant differences ($p > 0.05$) were observed between oils extracted from control and heated walnut paste after 20 days of storage at 35 °C, indicating that the heat treatment temperature and time selected for enzyme inactivation had little influence on FFA values. But there was a significant effect of blanching treatment at 120 °C for 15 min on the FFA values of hazelnut meal, resulting in the increased FFA values with storage time (Cam & Kilic, 2009). Various effects on FFA values could be probably caused by different treatment/storage temperature and time. FFA value less than 0.6 g/100 g indicate acceptable walnut quality walnuts recommended by walnut industry in California, USA (Wang et al., 2007) even after heat treatments at 90 °C for 15 min followed by 20 d of accelerated storage at 35 °C. The results described above are in good agreement with those reported by Buranasompob et al. (2003) and Wang et al. (2006), in which FFA values of heated walnut kernels increase with increasing storage time.

Fig. 3 shows the GC profile of the FA from certified standard mixes (a) and walnut paste oil of control sample at 0 storage day (b). Fatty acid data of the walnut paste oils determined by GC are listed in Table 5. Immediately after heating (0 storage day), the major monounsaturated fatty acid (MUFA) present in walnut paste was oleic acid (C18:1), which ranged from 21.38 to 23.84 g/100 g. Meanwhile, linoleic acid (C18:2) was the most abundant polyunsaturated fatty acid (PUFA) at 57.04–60.11 g/100 g with a less

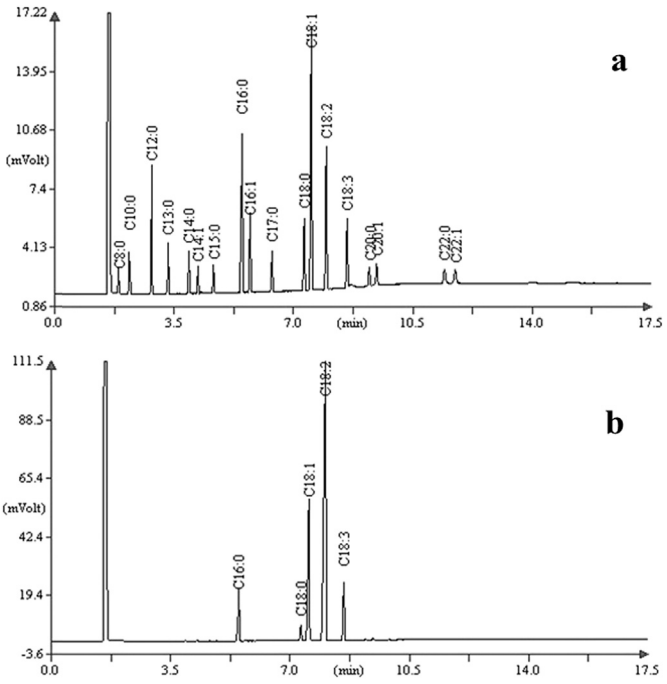

Fig. 3. GC chromatogram of (a) certified standard mixes and (b) walnut oil of control sample at 0 storage day. GC column and conditions could be found in materials and methods.

Table 5
Fatty acid composition (relative g/100 g) of oil extracted from walnut paste with different heating during accelerated storage.

Fatty acid	Storage time (*d*)	Treatment									
		Control	60 °C/10 min	60 °C/20 min	60 °C/30 min	75 °C/10 min	75 °C/15 min	75 °C/20 min	90 °C/5 min	90 °C/10 min	90 °C/15 min
16:0	0	7.51 ± 0.2	6.93 ± 0.2	6.64 ± 0.1	6.53 ± 0.2	6.42 ± 0.2	7.39 ± 0.4	6.62 ± 0.1	6.60 ± 0.2	7.58 ± 0.1	6.42 ± 0.2
	10	6.43 ± 0.1	6.52 ± 0.3	6.40 ± 0.2	6.38 ± 0.3	6.55 ± 0.1	6.51 ± 0.3	6.68 ± 0.2	6.58 ± 0.2	6.42 ± 0.1	6.56 ± 0.0
	20	6.48 ± 0.3	7.01 ± 0.1	6.79 ± 0.1	6.53 ± 0.1	6.82 ± 0.2	6.62 ± 0.2	6.56 ± 0.0	6.24 ± 0.4	6.08 ± 0.2	6.55 ± 0.2
18:0	0	1.82 ± 0.1	1.73 ± 0.0	1.56 ± 0.0	1.42 ± 0.0	1.60 ± 0.1	1.42 ± 0.0	1.47 ± 0.0	1.58 ± 0.0	1.41 ± 0.0	1.43 ± 0.0
	10	1.29 ± 0.0	1.43 ± 0.1	1.38 ± 0.0	1.47 ± 0.1	1.39 ± 0.1	1.27 ± 0.1	1.47 ± 0.0	1.41 ± 0.0	1.38 ± 0.1	1.32 ± 0.0
	20	1.43 ± 0.1	0.99 ± 0.0	1.32 ± 0.0	1.32 ± 0.0	1.27 ± 0.0	1.22 ± 0.0	1.20 ± 0.0	1.23 ± 0.0	1.20 ± 0.0	1.35 ± 0.0
18:1	0	22.76 ± 1.2	23.82 ± 0.6	23.84 ± 0.7	22.53 ± 1.6	23.14 ± 0.2	22.48 ± 0.9	22.30 ± 0.3	22.88 ± 0.1	21.44 ± 1.0	21.38 ± 0.2
	10	21.48 ± 0.9	21.89 ± 1.1	21.62 ± 0.5	20.72 ± 1.1	18.94 ± 0.1	18.88 ± 0.1	19.41 ± 0.5	20.42 ± 0.3	20.55 ± 0.6	18.99 ± 0.6
	20	20.97 ± 0.9	20.73 ± 0.3	21.2 ± 0.1	19.74 ± 0.2	20.94 ± 0.8	19.71 ± 0.4	20.61 ± 1.2	22.16 ± 0.8	20.52 ± 0.3	19.04 ± 0.3
18:2	0	59.25 ± 1.8	57.04 ± 0.4	57.95 ± 0.7	57.38 ± 0.5	57.99 ± 1.1	60.60 ± 0.1	60.11 ± 0.2	58.35 ± 0.1	59.11 ± 1.2	59.03 ± 0.6
	10	58.90 ± 1.1	56.82 ± 0.2	57.38 ± 0.1	58.61 ± 1.4	58.51 ± 0.5	59.91 ± 0.3	58.89 ± 0.5	57.96 ± 0.9	57.91 ± 0.7	58.61 ± 0.3
	20	57.02 ± 0.7	58.52 ± 0.5	57.09 ± 0.5	59.21 ± 0.9	57.47 ± 0.2	59.79 ± 0.4	58.55 ± 0.3	57.48 ± 0.4	57.12 ± 0.1	59.92 ± 1.0
18:3	0	10.65 ± 0.1	10.49 ± 0.3	10.21 ± 0.9	9.14 ± 0.2	10.86 ± 0.5	9.51 ± 0.1	9.51 ± 0.1	10.59 ± 0.2	8.86 ± 0.6	9.74 ± 0.8
	10	11.90 ± 0.2	13.34 ± 0.5	13.21 ± 0.6	12.82 ± 0.1	14.61 ± 0.4	13.43 ± 0.3	13.75 ± 0.0	13.63 ± 0.1	13.74 ± 0.2	14.52 ± 0.6
	20	14.10 ± 0.1	12.75 ± 0.1	13.60 ± 0.2	13.21 ± 0.4	13.50 ± 0.1	12.65 ± 0.3	13.08 ± 0.6	12.90 ± 0.4	15.08 ± 0.5	13.41 ± 0.1
PUFA	0	69.90 ± 1.1	67.53 ± 0.2	68.16 ± 0.4	66.52 ± 0.3	68.85 ± 0.7	70.11 ± 0.2	69.62 ± 0.1	68.94 ± 0.2	67.97 ± 0.4	68.77 ± 0.4
	10	70.80 ± 0.5	70.16 ± 0.3	70.59 ± 0.2	71.43 ± 0.8	73.12 ± 0.3	73.34 ± 0.2	72.64 ± 0.2	71.59 ± 0.3	71.65 ± 0.4	73.13 ± 0.3
	20	71.12 ± 0.3	71.27 ± 0.2	70.69 ± 0.1	72.42 ± 0.6	70.97 ± 0.2	72.44 ± 0.2	71.63 ± 0.5	70.38 ± 0.3	72.20 ± 0.1	73.33 ± 0.4

16:0: Palmitic acid, 18:0: Stearic acid, 18:1: Oleic acid, 18:2: Linoleic acid, 18:3: Linolenic acid, PUFA: Linoleic acid + Linoleic acid.

amount of linolenic acid (C18:3) at 8.86–10.86 g/100 g. The primary saturated fatty acids (SFA) identified in walnuts were palmitic acid (C16:0) and stearic acid (C18:0), which had 6.42–7.58 and 1.41–1.82 g/100 g, respectively. Overall, the concentrations of total SFA, MUFA and PUFA remained unchanged for oils extracted from different heated walnut paste, suggesting that heating had little effect on the fatty acid composition of the oils tested, which was probably caused by the relatively stable fatty acids in the walnut oil. These unchanged FA compositions under different heating temperature and time combinations were also reported for rice germ and safflower seed oils (Chandrasekara & Shahidi, 2011; Kim et al., 2002; Lee, Oh, Chang, & Kim, 2004;).

On the contrary, the concentration of stearic acid (C18:0) and oleic acid (C18:1) showed slight decreases during storage, while the concentration of linolenic acid (C18:3) and PUFA clearly increased with storage time. However there was no significant difference ($p > 0.05$) among heated samples during storage (Table 5).

3.4. Comparison between walnut kernel paste and oils with different packages during storage

Fig. 4 shows the mean PV, CDV, and FFA value of three different walnut products with different packages over the 20 d of accelerated storage at 35 °C. The mean PV of oils extracted from vacuum packaged walnut paste decreased with increasing storage time, while that for oils extracted from normally packaged walnut paste remained almost constant during storages. After 20 d of storage the mean PV of oils extracted from two types packaged walnut paste were 0.22 and 0.40 meq/kg, respectively. The mean PV of oils extracted from vacuum packaged walnut kernels did not vary significantly ($p > 0.05$) over storage period, but that for oils extracted from normally packaged walnut kernels increased with increasing storage time. After 20 d of storage, the mean PV of oils extracted from walnut kernels with normal and vacuum packages were 0.47 and 0.85 meq/kg, respectively. In contrast, the mean PV

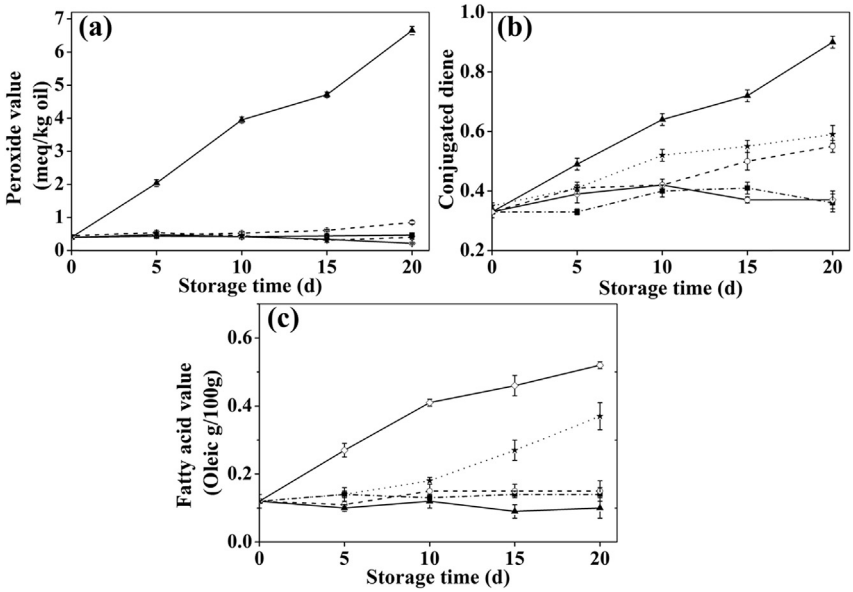

Fig. 4. Changes of (a) peroxide values, (b) conjugated diene values and (c) fatty acid values of oil from unheated walnut products with different packages during accelerated storage at 35 °C (■Kernel in vacuum package ○Kernel in glass beakers ▲Oil in screw-cap test tubes ◇Paste in vacuum package ★Paste in glass beakers).

of cold-pressed walnut oil in screw-cap tubes was significantly higher ($p < 0.05$) than that in walnut kernels and paste during storage. After 5, 10, 15, and 20 d of storage at 35 °C, the PV increased sharply and reached 2.04, 3.95, 4.71, and 6.65 meq/kg of oil, respectively. The mean CDV of oils extracted from walnut kernels and paste packaged in normal or cold-pressed walnut oils in screw-cap tubes increased gradually as the storage time increased and reached 0.55, 0.59 and 0.90, respectively, after 20 d of storage. The CDV of oils extracted from walnut kernels and paste packaged in vacuum remained almost constant during storages, which were 0.36 and 0.37, respectively, after 20 d of storage.

Data from this experiment indicate that the walnut paste is more stable against oxidation than walnut kernels and cold-pressed walnut oils, even after 20 d of accelerated storage at 35 °C with normal package. As the walnut paste has a large surface area and damaged structure, it might have been expected that deterioration reactions may take place fast compared with intact kernel, particularly as it contains high levels of PUFA. Arranz, Perez-Jimenez, and Saura-Calixto (2008) reported that the defatted matter flour provided the bulk of the antioxidant capacity of walnut, a major proportion derived from insoluble tannins, which are located mainly in the pellicle that separates the nut kernels. Arcan and Yemenicioğlu (2009) reported removal of pellicle reduced the total antioxidant activity of walnut kernels by almost 90%. Samaranayaka, John, and Shahidi (2008) reported walnut phenolics, similar to other seeds, grains and nuts, may be expected to reside in highest concentration in the pellicle of the nut. It is possible that the oil in the walnut paste only remained stable because the phenolic compounds present in the pellicle are active in paste oils but not in cold pressed oils. However, Vanhanen and Savage (2006) indicate that the residual oil contained in ground walnut flour is more stable than commercial walnut oil stored in sealed bottles since the flour contains high levels of vitamin E.

Regarding FFA values, two types packaged walnut kernels and walnut oils in screw-cap tubes did not vary significantly along storage period, which were 0.14, 0.15 and 0.10%, respectively, after 20 d of storage. In contrast, the FFA value of oils extracted from two selected packaged walnut paste increased with increasing storage time and were 0.52 and 0.37%, respectively, after 20 d of storage. This implies that the oils contained in walnut paste are more likely to hydrolytic rancidity than those in walnut kernels and cold-pressed oils, especially for vacuum packaged paste. Walnut paste may be effectively used as an ingredient for bakery products and for many frequently consumed foods (Ayo et al., 2007; ErcoŞKun & Demirci-ErcoŞKun, 2010; Gómez, Oliete, Caballero, Ronda, & Blanco, 2008; Oliete et al., 2008). The results observed in this experiment demonstrate that FFA values in walnut paste should be used as a key parameter for walnut quality evaluation during storage.

4. Conclusions

Thermal treatment for enzyme inactivation had an adverse effect on the initial quality of walnut paste, but still provided acceptable quality recommended by the US walnut industry. The PV and CDV of the walnut paste were reduced or stable, while the FFA values increased during storage time. After 20 days of storage, however, the oil quality in all heated walnut paste was not changed significantly. Walnut paste with normal or vacuum package was more stable than walnut kernels and cold-pressed walnut oils during storage. Since walnut paste was prone to hydrolytic rancidity, the FFA value significantly increased during storage and was the major parameter to limit long-term storage of walnut paste in commercial practices. The storage experimental results showed that walnut paste kept in vacuum or normal package at ambient temperature (25 °C) could be easily stored for up to two months with entirely satisfactory quality.

Acknowledgments

This research was supported by research grants from General Program of National Natural Science Foundation of China (No. 31371853) and Ph.D. Programs Foundation of Ministry of Education of China (20120204110022).

References

AOAC. (1997). *Official methods of analysis of the Association of Official Analytical Chemists*. Champaign: Illinois.
Arcan, I., & Yemenicioğlu, A. (2009). Antioxidant activity and phenolic content of fresh and dry nuts with or without the seed coat. *Journal of Food Composition and Analysis, 22*(3), 184–188.
Arena, E., Campisi, S., Fallico, B., & Maccarone, E. (2007). Distribution of fatty acids and phytosterols as a criterion to discriminate geographic origin of pistachio seeds. *Food Chemistry, 104*(1), 403–408.
Arranz, S., Perez-Jimenez, J., & Saura-Calixto, F. (2008). Antioxidant capacity of walnut (*Juglans regia* L.): contribution of oil and defatted matter. *European Food Research and Technology, 227*(2), 425–431.
Ayo, J., Carballo, J., Serrano, J., Olmedilla-Alonso, B., Ruiz-Capillas, C., & Jiménez-Colmenero, F. (2007). Effect of total replacement of pork backfat with walnut on the nutritional profile of frankfurters. *Meat Science, 77*(2), 173–181.
Ayo, J., Carballo, J., Solas, M. T., & Jiménez-Colmenero, F. (2005). High pressure processing of meat batters with added walnuts. *International Journal of Food Science & Technology, 40*(1), 47–54.
Bakkalbasi, E., Yilmaz, O. M., Javidipour, I., & Artik, N. (2012). Effects of packaging materials, storage conditions and variety on oxidative stability of shelled walnuts. *LWT–Food Science and Technology, 46*(1), 203–209.
Breda, C. A., Sanjinez-Argandoña, E. J., & Correia, C.d. A. C. (2012). Shelf life of powdered Campomanesia adamantium pulp in controlled environments. *Food Chemistry, 135*(4), 2960–2964.
Buranasompob, A., Tang, J., Mao, R., & Swanson, B. G. (2003). Rancidity of walnuts and almonds affected by short time heat treatments for insect control. *Journal of Food Processing and Preservation, 27*(6), 445–464.
Cai, L., Cao, A., Aisikaer, G., & Ying, T. (2013). Influence of kernel roasting on bioactive components and oxidative stability of pine nut oil. *European Journal of Lipid Science and Technology, 115*(5), 556–563.
Cam, S., & Kilic, M. (2009). Effect of blanching on storage stability of hazelnut meal. *Journal of Food Quality, 32*(3), 369–380.
Chandrasekara, N., & Shahidi, F. (2011). Oxidative stability of cashew oils from raw and roasted nuts. *Journal of the American Oil Chemists' Society, 88*(8), 1197–1202.
Cofrades, S., Serrano, A., Ayo, J., Carballo, J., & Jiménez-Colmenero, F. (2008). Characteristics of meat batters with added native and preheated defatted walnut. *Food Chemistry, 107*(4), 1506–1514.
Crews, C., Hough, P., Godward, J., Brereton, P., Lees, M., Guiet, S., et al. (2005). Study of the main constituents of some authentic walnut oils. *Journal of Agricultural and Food Chemistry, 53*(12), 4853–4860.
Crowe, T. D., & White, P. J. (2003). Oxidative stability of walnut oils extracted with supercritical carbon dioxide. *Journal of the American Oil Chemists Society, 80*(6), 575–578.
ErcoŞKun, H., & Demirci-ErcoŞKun, T. (2010). Walnut as fat replacer and functional component in sucuk. *Journal of Food Quality, 33*(5), 646–659.
FAOSTAT. (2012). *Food and Agriculture Organization*. Available at http://faostat.fao.org/site/339/default.aspx.
Gómez, M., Oliete, B., Caballero, P., Ronda, F., & Blanco, C. (2008). Effect of nut paste enrichment on wheat dough rheology and bread volume. *Food Science and Technology International, 14*(1), 57–65.
Gao, M., Tang, J., Wang, Y., Powers, J., & Wang, S. (2010). Almond quality as influenced by radio frequency heat treatments for disinfestation. *Postharvest Biology and Technology, 58*(3), 225–231.
Kim, I.-H., Kim, C.-J., You, J.-M., Lee, K.-W., Kim, C.-T., Chung, S.-H., et al. (2002). Effect of roasting temperature and time on the chemical composition of rice germ oil. *Journal of the American Oil Chemists' Society, 79*(5), 413–418.
Kong, F. B., Tang, J. M., Rasco, B., & Crapo, C. (2007). Kinetics of salmon quality changes during thermal processing. *Journal of Food Engineering, 83*(4), 510–520.
Labuckas, D., Maestri, D., & Lamarque, A. (2011). Lipid and protein stability of partially defatted walnut flour (*Juglans regia* L.) during storage. *International Journal of Food Science and Technology, 46*(7), 1388–1397.
Laskawy, G., Senser, F., & Grosch, W. (1983). Storage stability of roasted hazelnuts. *Review for Chocolate, Confectionery and Bakery, 8*(3), 21–23.
Lee, Y.-C., Oh, S.-W., Chang, J., & Kim, I.-H. (2004). Chemical composition and oxidative stability of safflower oil prepared from safflower seed roasted with different temperatures. *Food Chemistry, 84*(1), 1–6.
Lopez, A., Pique, M., Romero, A., & Aleta, N. (1995). Influence of cold-storage conditions on the quality of unshelled walnuts. *International Journal of Refrigeration, 18*(8), 544–549.

Martinez, M., Barrionuevo, G., Nepote, V., Grosso, N., & Maestri, D. (2011). Sensory characterisation and oxidative stability of walnut oil. *International Journal of Food Science and Technology, 46*(6), 1276–1281.
Martinez, M. L., & Maestri, D. M. (2008). Oil chemical variation in walnut (*Juglans regia* L.) genotypes grown in Argentina. *European Journal of Lipid Science and Technology, 110*(12), 1183–1189.
Mate, J. I., Saltveit, M. E., & Krochta, J. M. (1996). Peanut and walnut rancidity: effects of oxygen concentration and relative humidity. *Journal of Food Science, 61*(2), 465–469.
Mcglamery, J. B., & Hood, M. P. (1951). Effect of two heat treatments on rancidity development in unshelled pecans. *Journal of Food Science, 16*(1–6), 80–84.
Mexis, S. F., Badeka, A. V., Riganakos, K. A., Karakostas, K. X., & Kontominas, M. G. (2009). Effect of packaging and storage conditions on quality of shelled walnuts. *Food Control, 20*(8), 743–751.
Mitcham, E. J., Veltman, R. H., Feng, X., de Castro, E., Johnson, J. A., Simpson, T. L., et al. (2004). Application of radio frequency treatments to control insects in in-shell walnuts. *Postharvest Biology and Technology, 33*(1), 93–100.
Mitchell, J. H., & Malphrus, R. K. (1977). Lipid oxidation in spanish peanuts: the effect of moist heat treatments. *Journal of Food Science, 42*(6), 1457–1461.
Nayak, B., Berrios, J. D. J., Powers, J. R., & Tang, J. M. (2011). Thermal degradation of anthocyanins from purple potato (cv. purple majesty) and impact on antioxidant capacity. *Journal of Agricultural and Food Chemistry, 59*(20), 11040–11049.
Oliete, B., Gómez, M., Pando, V., Fernández-Fernández, E., Caballero, P., & Ronda, F. (2008). Effect of nut paste enrichment on physical characteristics and consumer acceptability of bread. *Food Science and Technology International, 14*(3), 259–269.
Ovissipour, M., Rasco, B., Tang, J., & Sablani, S. S. (2013). Kinetics of quality changes in whole blue mussel (*Mytilus edulis*) during pasteurization. *Food Research International, 53*(1), 141–148.
Piccirillo, P., Fasano, P., Mita, G., De Paolis, A., & Santino, A. (2006). Exploring the role of lipoxygenases on walnut quality and shelf-life. In M. E. Malvolti, & D. Avanzato (Eds.), *Proceedings of the Fifth International Walnut Symposium* (pp. 543–545). Leuven 1: International Society Horticultural Science.
Piffaut, B., & Metche, M. (1991). Properties of peroxidase and polyphenol oxidase in natural complexes from walnuts (*Juglans regia*) and in active DL-DOPA copolymers. *Journal of the Science of Food and Agriculture, 57*(4), 493–506.
Samaranayaka, A. G. P., John, J. A., & Shahidi, F. (2008). Antioxidant activity of English walnut (*Juglans regia* L.). *Journal of Food Lipids, 15*(3), 384–397.
Senter, S., Forbus, W., Nelson, S., Wilson, R., & Horvat, R. (1984). Effects of dielectric and steam heating treatments on the storage stability of pecan kernels. *Journal of Food Science, 49*(3), 893–895.
Shimoda, H., Tanaka, J., Kikuchi, M., Fukuda, T., Ito, H., Hatano, T., et al. (2008). Walnut polyphenols prevent liver damage induced by carbon tetrachloride and D-Galactosamine: hepatoprotective hydrolyzable tannins in the kernel pellicles of walnut. *Journal of Agricultural and Food Chemistry, 56*(12), 4444–4449.
Taoukis, P. S., Labuza, T. P., & Saguy, I. S. (1997). *Kinetics of food deterioration and shelf-life prediction*. New York: CRC Press.
Vanhanen, L. P., & Savage, G. P. (2006). The use of peroxide value as a measure of quality for walnut flour stored at five different temperatures using three different types of packaging. *Food Chemistry, 99*(1), 64–69.
Venkatachalam, M., & Sathe, S. K. (2006). Chemical composition of selected edible nut seeds. *Journal of Agricultural and Food Chemistry, 54*(13), 4705–4714.
Wang, S., Monzon, M., Johnson, J. A., Mitcham, E. J., & Tang, J. (2007). Industrial-scale radio frequency treatments for insect control in walnuts: II: Insect mortality and product quality. *Postharvest Biology and Technology, 45*(2), 247–253.
Wang, S., Tang, J., Sun, T., Mitcham, E. J., Koral, T., & Birla, S. L. (2006). Considerations in design of commercial radio frequency treatments for postharvest pest control in in-shell walnuts. *Journal of Food Engineering, 77*(2), 304–312.
Watkins, C. (2005). The world of Nuts-Often maligned for their high fat content, nuts are actually a good source of phytochemicals. *Inform-International News on Fats Oils and Related Materials, 16*(4), 200–203.
Yeşiloğlu, Y., & Demirkan, B. (2010). Biocatalytic properties of lipase from walnut seed (*Juglans regia* L.). *Journal of the American Oil Chemists' Society, 87*(6), 659–665.
Zacheo, G., Cappello, M., Gallo, A., Santino, A., & Cappello, A. (2000). Changes associated with post-harvest ageing in almond seeds. *LWT–Food Science and Technology, 33*(6), 415–423.
Zwarts, L., Savage, G. P., & Mcneil, D. L. (1999). Fatty acid content of New Zealand-grown walnuts (*Juglans regia* L.). *International Journal of Food Sciences and Nutrition, 50*(3), 189–194.