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Determination of fatty acid composition of γ-irradiated hazelnuts, walnuts, almonds, and pistachios

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ABSTRACT

Hazelnut, walnut, almonds, and pistachio nuts were treated with 1, 3, 5, and 7 kGy of gamma irradiation, respectively. Oil content, free fatty acid, peroxide value, and fatty acid composition of the nuts were investigated immediately after irradiation. The data obtained from the experiments indicated that gamma irradiation did not cause any significant change in the oil content of nuts. In contrast, free fatty acid and peroxide value of the nuts increased proportionally to the dose ($p<0.05$). Among the fatty acids determined, the concentration of total saturated fatty acids increased while total monounsaturated and total polyunsaturated fatty acids decreased with the irradiation dose ($p<0.05$ and <0.01).

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1. Introduction

Consumption of nuts has seen an upward trend in Turkey where, in 2009, it has been estimated at approximately 3 kg/capita, while this rate was less than 1 kg/capita in the world. Nuts are eaten raw or roasted; they are significant components in a variety of traditional dishes in our country. In 2009, the production amount of all nut types in Turkey alone was about 300,000 ton; exports of these nuts were worth an approximately $500 million (Anonymous, 2010).

The extension of shelf life is the main purpose of radiation processing of foods (Golge and Ova, 2008). Irradiation has become an effective means of processing and preserving food products and an effective way of inactivating food-borne pathogens in foods (Gumus et al., 2008; Thomas et al., 2008). Food irradiation is a process in which products are exposed to radiant energy containing gamma rays, electron beams, and X-rays at the doses specified by the Food and Drug Administration (Yilmaz and Gecgel, 2007). Among these three irradiation applications, γ-irradiation has been preferred in previous researches in post-harvest pest control and in controlling insect infestation in many agricultural products, for example, for fruits such as kiwis, apples, apricots, coconuts, figs, dates, pears, berries, mangoes, raisins, and prunes; for tree nuts such as cashew nuts, almonds, and walnuts; and for onions, potatoes, carrots, beetroots, rice, ginger, and garlic (Bhattacharjee et al., 2003; Al-Bachir, 2004; Nayak et al., 2007; Perez et al., 2007; Kim and Yook, 2009; Mexis and Kontominas, 2009; Mexis et al., 2009; Kim et al., 2010). High doses of radiation can sometimes bring about undesirable changes in the flavor, appearance, and texture of food, culminating in a product unfit for human consumption (Sanchez-Bel et al., 2005). For irradiation of food stuffs, maximum overall dose of 10 kGy is permitted in numerous countries for commercial food processing (Lacroix and Quattara, 2000). However, nuts contain high levels of unsaturated fatty acids that are prone to lipid peroxidation when irradiated (Mexis et al., 2009). Furthermore, in unsaturated fatty acids, radiolysis generally brings about loss of essential acids with nutritional impairment (Mexis and Kontominas, 2009). Irradiation also produces free radicals, esters, ketones, sulfur compounds, and aldehydes as a result of radiolysis (Sajilata and Singhal, 2006). Such compounds in order are responsible for changes of flavor in irradiated foods (Diehl, 1981). Therefore, it is highly significant to study the effect of γ-irradiation on some chemical characteristics of nuts. The objective of the present study was to determine the effect of gamma irradiation on oxidative stability and fatty acid composition of some nuts at irradiated doses of 1, 3, 5, and 7 kGy.

2. Materials and methods

2.1. Materials

2.1.1. Preparation of nut samples

In this study, samples of hazelnut (*Corylus avellana* L.-Giresun variety, without shell), walnut (*Juglans regia* L.-Şebin variety, without shell), almond (*Prunus dulcis*-Akbadem variety, without shell), and pistachio (*Pistacia vera* L.-Anaçıtlak variety, without shell) nuts were purchased from local markets in Tekirdag province in Turkey. All samples (each package is 250 g) were transferred into 60-gauge thick self-sealable low-density polyethylene (LDPE)

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pouches for irradiation. All samples were kept in a refrigerator (±4 °C) until irradiation was performed.

2.1.2. Irradiation of the nuts

All samples were immediately transported with a cooling box at 4 °C for irradiation treatments. The samples were irradiated at the GAMMAPAK Company, Cerkezkoy, Tekirdag, Turkey. The irradiation process of the samples was carried out in ^{60}Co gamma irradiator (MDS Nordion, Canada) at the average absorbed doses of 1, 3, 5, and 7 kGy for exposure times of 52, 156, 260, and 364 min, respectively. The absorbed dose was monitored by a Harwell Amber Perspex dosimeter.

2.1.3. Oil extraction

Proximate composition was evaluated immediately after irradiation. For chemical analysis, each sample of hazelnut, walnut, almond, and pistachio nuts was ground (particle size ≤0.5 mm) in a mill. The samples were homogenized by mixer and later analyzed to determine oil content. Lipid extraction from the nut samples was carried out by hexane extraction under the operating conditions specified in IUPAC methods no. 1.121, and expressed as a percentage by mass of the product as received (IUPAC, 1987). The samples were analyzed in triplicate, and then the mean was calculated. The obtained oil was stored at 4 °C for further investigation.

2.1.4. Free fatty acids

Free fatty acids (FFA) were measured by direct titration of the nuts' oil extract with (0.1 N) NaOH using phenolphthalein as an indicator. Free fatty acid contents of oil samples were determined in accordance with methods no. 2.201 of IUPAC (1987).

2.1.5. Peroxide value

The peroxide value (PV) was determined using the extracted oil and estimated as the iodine was released as a product of the oxidation of potassium iodide by the peroxides, or other similar products of oil oxidation. The value acquired was expressed as milliequivalents of O_2/kg of seed; the procedure was fulfilled according to methods no. 2.501 of IUPAC (1987).

2.1.6. Fatty acid methyl esters (FAMEs)

Fatty acid composition was determined by gas chromatography. The fatty acid composition was determined on the lipid extracts after methylation to form fatty acid methyl esters (FAME) (AOAC, 1997), which were analyzed by using a Hewlett Packard 5890 Gas Chromatograph equipped with Supelco capillary column (100 m × 0.25 mm i.d., 0.20 μm film thickness) and flame ionization detector (FID). Hydrogen was used as the carrier gas. Thermal gradient ranged from 170 to 220 °C at 1 °C/min. Injector and FID temperature was 220 °C. Flow rate was 5 mL/min.

2.1.7. Statistical analysis

The data obtained from three replications were analyzed by ANOVA using the SPSS (version 10.0) statistical package program, and differences among the means were compared using the Duncan's multiple range test.

3. Results and discussion

Changes in oil content, FFA, and PV of nuts as functions of irradiation dose were shown in Table 1.

Results showed that there were no significant differences in oil content between irradiated and unirradiated nuts. These results are in agreement with previous work on the effect of gamma irradiation on other similar products. Inayatullah et al. (1987) did not detect significant changes in the contents of water, fat, ash, and carbohydrate after irradiation of soybeans with doses of 0.25, 0.5, 1.0, 2.5, and 5 kGy.

Table 1
Oil content, free fatty acid, and peroxide value of irradiated nuts.[a]

Samples	Irradiation doses (kGy)	Oil content (%)	Free fatty acid (oleic acid, %)	Peroxide value (meq O_2/kg)
Hazelnut	Control	65.63 ± 1.691	0.12 ± 0.011c	0.7 ± 0.115c
	1	66.38 ± 1.177	0.12 ± 0.005bc	0.8 ± 0.057c
	3	65.42 ± 1.755	0.13 ± 0.011bc	0.8 ± 0.023b
	5	66.47 ± 1.778	0.14 ± 0.011ab	1.5 ± 0.115b
	7	65.46 ± 1.200	0.16 ± 0.005a	1.6 ± 0.057a
	Irradiation	**NS**	*	*
Walnut	Control	69.20 ± 1.709	0.20 ± 0.005c	1.2 ± 0.230
	1	68.54 ± 1.177	0.22 ± 0.011bc	1.2 ± 0.577
	3	69.27 ± 1.766	0.22 ± 0.005bc	1.4 ± 0.230
	5	67.27 ± 2.274	0.24 ± 0.011ab	1.6 ± 0.346
	7	68.54 ± 1.709	0.26 ± 0.005a	1.8 ± 0.288
	Irradiation	**NS**	*	**NS**
Almond	Control	55.47 ± 1.703	0.28 ± 0.011d	0.8 ± 0.023c
	1	56.41 ± 1.737	0.30 ± 0.005cd	0.8 ± 0.017c
	3	54.45 ± 1.125	0.32 ± 0.011bc	1.2 ± 0.028b
	5	55.39 ± 1.766	0.34 ± 0.005ab	1.2 ± 0.011b
	7	56.57 ± 1.195	0.37 ± 0.011a	1.4 ± 0.034a
	Irradiation	**NS**	*	*
Pistachio	Control	60.37 ± 1.691	0.16 ± 0.011c	1.0 ± 0.115c
	1	61.84 ± 1.177	0.18 ± 0.005bc	1.0 ± 0.057c
	3	60.45 ± 1.755	0.18 ± 0.011bc	1.5 ± 0.023b
	5	61.52 ± 1.778	0.20 ± 0.011ab	1.5 ± 0.115b
	7	60.61 ± 1.200	0.23 ± 0.005a	1.8 ± 0.057a
	Irradiation	**NS**	*	*

NS—not significant.

* Significant at $p < 0.05$.

[a] Each value is an average of three determinations, and values in the same column with different letters show statistically significant differences.

FFA, which indicates hydrolysis and PV, which reveals oxidation in nuts, increased significantly ($p < 0.05$) with increasing irradiation dose. These statistical differences are clear in Table 1, which shows an increase in FFA and PV during irradiation treatments. For example, FFA went from 0.20% to 0.26% in walnuts. The FFA of irradiated walnuts with 0, 0.5, 1.0, 1.5, and 2.0 kGy of gamma irradiation was found to be from 0.66% to 2.02% by Al-Bachir (2004) and our results are in accordance with the results of this study. It is reported that the increasing FFA in nuts immediately after irradiation may be attributed to the degradation of large lipid molecules producing smaller molecules containing free fatty acids. Likewise, as shown in Table 1, PV increases from an initial value of 0.7 to 1.6 meq O_2/kg in hazelnuts after irradiation at a dose of 7 kGy. A dose of 7 kGy culminated in an approximately two-fold increase in PV. It is clear that increasing doses of irradiation enhanced lipid oxidation leads to hydroperoxides formation. Similarly, Golge and Ova (2008) reported that gamma rays interact with fat molecules to cause oxidation, decarboxilation, dehydration, and polymerization reactions giving rise to lipid oxidation. Moreover, irradiation is known to produce free radicals, and antioxidants from foods have been shown to scavenge free radicals (Mexis and Kontominas, 2009).

These effects of gamma irradiation on the oxidation of nuts immediately after irradiation are in agreement with those of Inayatullah et al. (1987) who reported that the PV of soybeans was significantly increased by irradiation with 0.25, 0.5, 1.0, 2.5, and 5 kGy. Similarly, Chiou (1994) found that the PV of peanut oils extracted from irradiated peanuts increased with increased irradiation dosage (2.5, 5.0, 7.5, and 10 kGy). Sanchez-Bel et al. (2005) indicated that immediately after irradiation there are significant differences in PV both in control and in irradiated almonds. Golge and Ova (2008) concluded that the peroxide value increases with the irradiation dose in pine nuts irradiated at doses between 0.5 and 5 kGy. Mexis et al. (2009) indicated that the PV of almonds was significantly increased (from 0.26 to 2.74 meqO2/kg) by irradiation with 1–7 kGy. In contrast, Jan et al. (1988) found that the treatment of shelled walnuts with 0.5 and 1 kGy did not affect lipid oxidation. These effects are in agreement with the findings of Al-Bachir (2004) who reported that immediately after irradiation there was no effects in peroxide value between irradiated and non-irradiated walnuts. Similarly, Sanchez-Bel et al. (2008) concluded that there was no change in PV after irradiation of almonds at doses 3, 7, and 10 kGy.

The fatty acid composition of oils extracted from the nuts processed with control, 1, 3, 5, and 7 kGy doses was given in Table 2.

Palmitic (C16:0), stearic (C18:0), oleic (C18:1), and linoleic (C18:2) acids were the principal fatty acids; pentadecanoic (C15:0), palmitoleic (C16:1), heptadesanoic (C17:1), arachidic (C20:0), eicosenoic (C20:1), docosahexaenoic (C22:6), and lignoceric (C24:0) acids were the minor fatty acids for all nuts analyzed. According to ANOVA, the effect of irradiation doses on C16:0, C18:0, C18:1, C18:2, total saturated fatty acids (SFA), total monounsaturated fatty acids (MUFA), total polyunsaturated fatty acids (PUFA), and total unsaturated fatty acids (UFA) contents was statistically significant ($p < 0.01$). The contents of C16:0 and C18:0 fatty acids increased parallel to the increase in irradiation doses but there was a decrease in C18:1 and C18:2 fatty acids. Likewise, the percentage of SFA increased with the irradiation treatments, whereas the percentage of MUFA and PUFA decreased. For example, the content of SFA, MUFA, and PUFA in control hazelnuts was 6.63%, 82.45%, and 10.92%, respectively. After irradiation at 7 kGy, the respective values were 8.88%, 81.46%, and 9.72%. C15:0, C16:0, C18:0, and C24:0 showed an increase after irradiation at doses > 1 kGy ($p < 0.05$ and < 0.01). The increase in C16:0 content after irradiation is very

Table 2
Fatty acid composition of irradiated nuts.[a]

Samples	Irradiation doses (kGy)	Fatty acids (%)															
		C15:0	C16:0	C16:1	C17:1	C18:0	C18:1	C18:2	C18:3	C20:0	C20:1	C22:6	C24:0	SFA	MUFA	PUFA	UFA
Hazelnut	Control	0.44	4.05d	0.12	0.07	2.02e	82.11a	10.75a	0.17	0.12	0.15	–	–	**6.63e**	**82.45a**	**10.92a**	**93.37a**
	1	0.42	4.28c	0.13	0.07	2.15d	82.09a	10.46b	0.14	0.11	0.15	–	–	**6.96d**	**82.44a**	**10.60b**	**93.04b**
	3	0.44	5.26b	0.13	0.07	2.29c	81.17c	10.24c	0.13	0.12	0.15	–	–	**8.11c**	**81.52c**	**10.37c**	**91.89c**
	5	0.45	5.28b	0.14	0.08	2.39b	81.68b	9.63d	0.12	0.11	0.12	–	–	**8.23b**	**82.02b**	**9.75d**	**91.77c**
	7	0.46	5.82a	0.14	0.08	2.47a	81.06d	9.60d	0.12	0.13	0.12	–	–	**8.88a**	**81.46d**	**9.72d**	**91.12d**
	Irradiation	**NS**	**	**NS**	**NS**	**	**	**	**NS**	**NS**	**NS**	–	–	**	**	**	**
Walnut	Control	0.52b	6.70e	0.05	–	2.58d	17.22e	59.83a	12.65a	0.09	0.18a	0.18c	–	**9.89e**	**17.45e**	**72.66a**	**90.11a**
	1	0.54ab	6.76d	0.05	–	2.59cd	17.70d	59.30b	12.61b	0.09	0.18a	0.18c	–	**9.98d**	**17.93d**	**72.09b**	**90.02b**
	3	0.54ab	6.83c	0.06	–	2.62c	18.13c	58.78c	12.59b	0.10	0.15ab	0.20c	–	**10.09c**	**18.34c**	**71.57c**	**89.91c**
	5	0.54ab	6.90b	0.06	–	2.94b	18.65b	58.13d	12.31c	0.10	0.13bc	0.24b	–	**10.48b**	**18.84b**	**70.68d**	**89.52d**
	7	0.56a	7.16a	0.05	–	3.20a	21.89a	55.11e	11.53d	0.11	0.11c	0.28a	–	**11.03a**	**22.05a**	**66.92e**	**88.97e**
	Irradiation	*	**	**NS**	–	**	**	**	**	**NS**	*	**	–	**	**	**	**
Almond	Control	–	6.75e	0.55	0.11	1.22c	64.62a	26.46a	0.07	–	–	–	0.22c	**8.19e**	**65.28a**	**26.53a**	**91.81a**
	1	–	7.09d	0.57	0.10	1.28b	64.55b	26.08c	0.07	–	–	–	0.26b	**8.63d**	**65.22b**	**26.15b**	**91.37b**
	3	–	7.21c	0.52	0.11	1.30b	64.57b	25.96d	0.07	–	–	–	0.26b	**8.77c**	**65.20b**	**26.03c**	**91.23c**
	5	–	7.34b	0.56	0.11	1.31b	64.23c	26.12b	0.05	–	–	–	0.28b	**8.93b**	**64.90c**	**26.17b**	**91.07d**
	7	–	7.85a	0.58	0.12	1.48a	63.95d	25.62e	0.05	–	–	–	0.35a	**9.68a**	**64.65d**	**25.67d**	**90.32e**
	Irradiation	–	**	**NS**	**NS**	**	**	**	**NS**	–	–	–	**	**	**	**	**
Pistachio	Control	–	7.75e	0.62a	–	1.95d	67.76b	20.73b	0.39a	–	0.74a	–	–	**9.70e**	**69.18a**	**21.12b**	**90.30a**
	1	–	8.00d	0.60a	–	2.08c	67.84a	20.53c	0.33b	–	0.62b	–	–	**10.08d**	**69.06b**	**20.86c**	**89.92b**
	3	–	8.61c	0.52b	–	2.16b	67.29c	20.86a	0.33b	–	0.23c	–	–	**10.77c**	**68.04c**	**21.19a**	**89.23c**
	5	–	9.05b	0.51b	–	2.24a	67.24d	20.45d	0.29c	–	0.22c	–	–	**11.29b**	**67.97d**	**20.74d**	**88.71d**
	7	–	10.18a	0.50b	–	2.26a	66.85e	19.73e	0.28c	–	0.20c	–	–	**12.44a**	**67.55e**	**20.01e**	**87.56e**
	Irradiation	–	**	**	–	**	**	**	**	–	**	–	–	**	**	**	**

UFA—total unsaturated fatty acids; NS—not significant.

* Significant at $p < 0.05$.

** Significant at $p < 0.01$.

[a] Each value is an average of three determinations, and values in the same column with different letters show statistically significant differences. Each value is expressed as weight wt% of total fatty acid methyl esters; SFA—total saturated fatty acids; MUFA—total monounsaturated fatty acids; PUFA—total polyunsaturated fatty acids.

important as experiments indicated that hamsters fed with diets high in C16:0 had raised plasma cholesterol concentrations (Mexis et al., 2009). The irradiated samples revealed a C18:1 content significantly lower than that of control samples; these differences were higher at the highest dose. Only irradiated walnut samples showed a C18:1 content higher than that of the control samples. The reason for this situation may be because the content of C18.3 is higher than in the other nuts such as hazelnuts, almonds, and pistachios. As can be seen from Table 2, the highest amount among nuts of C18:3 was found in walnuts (12.65%). According to Sanchez-Bel et al. (2005), a delay in the desaturation from C18:1 to C18:2 and C18:3 was caused by the treatment. With respect to polyunsaturated fatty acids, the contents of C18:2 in the irradiated samples were lower than those in the control samples. In addition, C18:3 showed a decrease after irradiation in both walnuts and pistachios ($p < 0.01$). The present results are in agreement with those of Mexis et al. (2009) who indicated significant changes ($p < 0.05$) in fatty acid composition of raw unpeeled almonds irradiated at 1, 1.5, 3, 5, and 7 kGy doses. It was concluded that the percentage of total saturated fatty acids increased (from 11.85% to 15.47%) with the irradiation doses, whereas the percentage of total monounsaturated (from 69.69% to 66.16%) and total polyunsaturated (from 18.46% to 18.37%) fatty acids decreased ($p < 0.05$). According to researchers, the radiolysis of fatty acids causes the formation of two groups of long chain hydrocarbons. The first group is comprised of hydrocarbons with one carbon less than the original fatty acid, the second group is comprised of hydrocarbons with two carbon atoms less than the original fatty acid and one additional double bond at position 1 by rupture of the side chain in the α- and β-positions with regard to the carbonyl group. Similarly, Mexis and Kontominas (2009) found that the effect of irradiation on C16:0, C18:0, C18:1, and C18:2 was statistically significant ($p < 0.05$) for cashew nuts. It is reported that there was a relative decrease in MUFA upon irradiation; PUFA was not affected by irradiation, and the relative percentage of SFA increased. The same researchers concluded that irradiation caused partial decomposition of a small number of triglycerides. Likewise, Sanchez-Bel et al. (2005) reported that the contents of saturated and unsaturated fatty acids changed with different irradiation doses. Researchers irradiated almonds with 3, 7, and 10 kGy and showed an increase in C16:0 (from 6.41% to 6.55%) and C18:0 (from 2.35% to 2.50%), and decrease in C18:2 (from 19.7% to 19.0%) with the irradiation dose.

4. Conclusions

In the present study oxidative stability and fatty acid composition of irradiated nuts at 1, 3, 5, and 7 kGy were compared to control nuts. FFA and PV significantly increased ($p < 0.05$) after irradiation. Also, the effect of irradiation doses on the oil content was statistically insignificant. There was a statistically significant difference ($p < 0.05$ and < 0.01) in fatty acid composition and SFA increases as the dose and MUFA, PUFA, and UFA decrease.

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