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## Characteristic Hydrocarbons and 2-Alkylcyclobutanones for Detecting $\gamma$ -Irradiated Sesame Seeds after Steaming, Roasting, and Oil Extraction

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Hydrocarbons and 2-alkylcyclobutanones in sesame seeds (*Sesamum indicum* L.) irradiated at 0.5–4 kGy were used to determine the effect of subsequent steaming, roasting, and oil extraction from the roasted samples on the changes in their concentrations. The concentrations of radiation-induced hydrocarbons increased almost linearly ( $R^2 = 0.8671-0.9953$ ) with the applied dose. The hydrocarbons, 1,7-hexadecadiene and 8-heptadecene, were detected only in the irradiated samples before and after three types of treatments at doses  $\ge 0.5$  kGy, but they were not detected in non-irradiated samples before and after treatment. These two hydrocarbons could be used as markers to identify irradiated sesame seeds. The concentrations of the three detected 2-alkylcyclobutanones, 2-dodecylcyclobutanone (2-DCB), 2-tetradecylcyclobutanone (2-TCB), and 2-(5'-tetradecenyl)cyclobutanone (2-TeCB), linearly increased with the irradiation dose. These compounds could be detected at doses  $\ge 0.5$  kGy but not in non-irradiated samples. The three types of treatments had no significant effect on the levels of 2-alkylcyclobutanones.

KEYWORDS: Sesame seeds; irradiation; detection; hydrocarbons; 2-alkylcyclobutanones; steaming; roasting; oil extraction

### INTRODUCTION

Sesame seed (*Sesamum indicum* L.) is one of the most important agricultural products for international trade and the oldest oilseed crop (*I*). Unlike other oil crops, sesame seed has been cultivated in Asia and Africa for its oil (49.7%) and protein (17.7%) (2). China, India, Myanmar, and Sudan are the primary producers of sesame seeds contributing to approximately 70% of its total world production (2). Major importing countries, including Korea, are the major source of edible oil for local consumption, and the importation of sesame seed from different countries has been steadily increasing (3, 4).

Most sesame seeds are consumed as roasted, extracted oil and steamed product. Furthermore, roasted sesame seed and extracted oil are widely used in the Eastern Asian countries (5)and traditionally used as condiments in many oriental foods, especially in Korea. In sesame seed, the characteristic aroma, color, and texture are developed during roasting treatment, which is the crucial step in sesame seed processing as in the case of other oil seeds and nuts (6).

Irradiation helps to ensure a safer and more plentiful food supply by extending shelf life and controlling pests and pathogens in food. Food irradiation is applied variously to provide decontamination of certain food additives (10-50 kGy)of spices, enzyme preparations, and ingredients, to improve technological properties (2-7 kGy) of food, to eliminate spoilage and pathogenic microorganisms (1-7 kGy), to extend shelf life (1-3 kGy), to delay physiological processes of fresh fruits and vegetables (0.25-1.0 kGy), to disinfest insects and parasites of food products (0.15-0.5 kGy), and to inhibit the sprouting of bulbs and tubers (0.05-0.15 kGy) (7). Food irradiation is commercialized in about 40 countries for various kinds of food products. Irradiation of sesame is permitted as an effective treatment for the disinfestations in Cuba at maximum 2 kGy (8).

The European Committee for Standardization (CEN) has now published 10 official protocols for the detection of irradiated foods; thus, identification of irradiated from non-irradiated foods is highly recommended to confirm both compliance with existing regulations and beneficial effects of irradiation treatment (9, 10). In lipid-containing foods, such as meat and oil seed, radiationinduced hydrocarbons and 2-alkylcyclobutanones can be detected by gas chromatography/mass spectrometry (GC/MS) analysis (11-13). These methods are regarded as the most suitable methods to detect whether fat-containing food has been irradiated or not. In this work, radiation-induced hydrocarbons and 2-alkylcyclobutanones in sesame seeds were used to determine

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the effect of subsequent steaming, roasting, and oil extraction from the roasted samples on the changes in their concentrations.

#### MATERIALS AND METHODS

Materials, Irradiation, and Preparation. Sesame seeds were purchased from local markets in Daegu, South Korea. Sesame seeds were packaged (2 kg in each pack) in low-density polyethylene (LDPE) film and irradiated at 0-4 kGy using a 100 kCi point source AECL, IR-79, cobalt-60  $\gamma$  irradiator (MDS Nordion International Co. Ltd., Ottawa, Ontario, Canada) at the Korean Atomic Energy Research Institute (KAERI), Daejon, South Korea. A ceric/cerous dosimeter (Harwell, U.K.) was used to confirm the total absorbed doses, and the error range was within  $\pm 5.6\%$ . In the first treatment, both irradiated and nonirradiated sesame seeds (2 kg) were steamed for 20 min in a boiling water bath (6). Roasting for the non-irradiated and irradiated samples (2 kg) was performed by heating at 220 °C with agitation (50 rpm) for 10 min (14) using a JIS-E04 electric roaster (JEIL Industrial Co. Ltd., Seoul, South Korea). Half of sesame seeds after roasting treatment were used for extraction of oil by a commercial pressing method from a local market. Control (non-irradiated) and irradiated samples before and after treatments (steaming, roasting, and oil extraction after roasting) were stored under dark conditions at refrigeration temperature (4  $\pm$  1 °C) until used.

Fatty Acid Analysis. The oil from the ground sesame seeds was extracted by blending 20 g of the sample, with sodium sulfate and 100 mL of *n*-hexane. The mixture was centrifuged for 20 min at 6000 rpm to isolate oil and passed through No. 41 Whatman filter paper. The solvent was completely concentrated with a rotary evaporator under vacuum and nitrogen gas. The extracted oil was analyzed for fatty acid profile and content and stored at -18 °C until use for hydrocarbon analysis. The triglycerides were then converted to their corresponding fatty acid methyl esters (FAMEs) using boron trifluoride/methanol (14%), according to the procedure described by Metcalf et al. (15).

Gas Chromatography–Flame Ionization Detector (GC–FID) Analysis. The FAMEs were analyzed on a Varian Star 3400 CX gas chromatograph (Varian Chromatography Group, CA), equipped with a FID. The column was a 30 m × 0.25 mm i.d., 0.25  $\mu$ m, DB-FFAP 122-3232 (J&W Scientific, Folsom, CA). The GC–FID conditions were as follows: helium carrier gas flow, 1 mL/min; injection volume, 1  $\mu$ L; injector temperatures, 240 °C; initial temperature, 150 °C, held for 2 min; 5 °C/min up to 180 °C, held for 5 min; 8 °C/min up to 240 °C, held for 29.5 min; detector temperature, 250 °C.

**Isolation of Hydrocarbons.** Hydrocarbon analysis involved oil extraction, column chromatography, and GC/MS identification by the EN 1784 method (*13*). Florisil (60–100 mesh) (Fisher Scientific, Pittsburgh, PA) was heated at 550 °C overnight to remove the volatile organic contaminants and cooled in a desiccator. It was then deactivated by addition of 3% (w/w) distilled water. The mixture was shaken for at least 20 min and stored for 10-12 h. Deactivated Florisil (30 g) was packed into a glass column ( $200 \times 23$  mm) with a Teflon stopcock. About 1 g of anhydrous sodium sulfate was added on the top of the Florisil column in a 1 cm layer, which was conditioned with 10 mL of *n*-hexane.

The extracted oil (1 g), stored at -18 °C, was mixed with 1 mL of *n*-eicosane (4 µg/mL of hexane) as an internal standard and then applied to the Florisil column and eluted with 60 mL of hexane at a flow rate of 3 mL/min. The eluted hexane was concentrated to a volume of 2 mL using a rotary vacuum evaporator and vacuum system (Buchi, B-180, Flawil, Switzerland) and further concentrated to a volume of 0.5 mL by nitrogen gas.

Isolation of 2-Alkylcyclobutanones. For all lipid-containing foods, including those with sesame seeds, radiation-induced 2-alkylcyclobutanones can be detected by GC/MS (*12*). The oil from the ground sesame seeds was extracted by blending 20 g of the sample with sodium sulfate and 100 mL of *n*-pentane/2-propanol (3:2, v/v). A Florisil column for alkylcyclobutanone analysis was prepared as described above. Extracted oil (0.2 g) was mixed with 1 mL of 2-cyclohexylcy-clohexanone (1  $\mu$ g/mL of hexane) as an internal standard, applied to the column, and eluted with 150 mL of hexane followed by 120 mL of diethylether/hexane (2:98, v/v), at a flow rate of 3 mL/min. This latter

Table 1. Composition of Main Fatty Acids in Sesame

fatty acids cont	tern (pecent of total latty acid)
linoleic acid (18:2 cis) oleic acid (18:1 cis) palmitic acid (16:0) stearic acid (18:0) others total	$\begin{array}{c} 49.3 \pm 0.20^{a} \\ 38.7 \pm 0.19 \\ 6.6 \pm 0.00 \\ 4.0 \pm 0.47 \\ 1.4 \pm 0.03 \\ 100 \end{array}$

<sup>a</sup> Mean  $\pm$  standard deviation (sd) (n = 3).

fraction was concentrated into a volume of 2 mL using a rotary vacuum evaporator and further concentrated into a volume of 0.2 mL by nitrogen gas.

GC/MS Analysis. The isolated hydrocarbons and 2-alkylcyclobutanones were analyzed by a 6890 model gas chromatograph (Hewlett-Packard, Wilmington, DE) and Agilent 5793N mass selective detector (Agilent Technologies, Inc., Palo Alto, CA) equipped with a mass spectrometric detector in the electron impact ionization (EI) mode. The column used was a 30 m  $\times$  0.32 mm i.d., 0.25  $\mu$ m DB-5 (J&W Scientific, Folsom, CA). The ionization voltage and ion source temperature were 70 eV and 250 °C, respectively.

In case of hydrocarbons, the oven temperature was programmed for 60-170 °C at 25 °C/min, 205 °C at 2 °C/min, and 270 °C at 10 °C/min. The temperatures of injector and detector were 250 °C. Helium was the carrier gas, at a flow rate of 1.0 mL/min. The sample (2  $\mu$ L) was injected in splitless mode for 2 min and then in split mode (1:10). Hydrocarbons were identified by comparing retention time and mass spectra of peaks from the total ion chromatograms to those of authentic standard hydrocarbons. Standard hydrocarbons purchased from Sigma Co. (Sigma Aldrich, St. Louis, MO) included 1-tetradecene (C<sub>14:1</sub>), pentadecane (C<sub>15:0</sub>), 1-hexadecene (C<sub>16:1</sub>), 1,7-hexadecadiene (C<sub>16:2</sub>), heptadecane (C<sub>17:2</sub>) and 8-hepatdecene (C<sub>16:3</sub>) did not confirm in this study because they could not be purchased.

In the case of 2-alkylcyclobutanones, the oven temperature was programmed for 120 °C (1 min), 160 °C at 15 °C/min, 175 °C at 0.5 °C/min, and 290 °C at 30 °C/min (10 min). The sample (2  $\mu$ L) was injected in splitless mode for 2 min and then in split mode (1:20). The quantitative analysis of 2-alkylcyclobutanones was set to selected ion monitoring (SIM) mode. The other conditions were the same as those of analysis. The standard purchased from Sigma Co. included 2-dode-cylcyclobutanone (DCB, C<sub>12:0</sub>), 2-(5'-tetradecenyl) cyclobutanone (2-TeCB, C<sub>14:1</sub>), and 2-tetradecylcyclobutanone (2-TCB, C<sub>14:0</sub>).

**Statistical Analysis.** The data were analyzed using the Microsoft Excel 2003 computer program. All measurements were performed 3 times (n = 3).

#### **RESULTS AND DISCUSSION**

**Fatty Acid Composition.** Moisture and crude fat contents of sesame seeds were 7.3 and 49.6%, respectively. The fatty acids in sesame seeds are palmitic, stearic, oleic, and linoleic acids. Linoleic acid (49.3%) was the most abundant fatty acid in sesame seeds, followed by oleic acids (38.7%) and small amounts of palmitic acid (6.6%) and stearic acid (4.2%) (**Table 1**). These results were similar to those reported by Abou-Gharbia et al. (6).

**Hydrocarbons Characteristics.** The radiolytic breakdown of triglycerides yields a series of saturated and unsaturated hydrocarbons, the type and quantity of which depend upon the glyceride fatty acid composition (*16*). Of the hydrocarbons produced from each irradiated fatty acid, mainly two types of hydrocarbons are formed. One has one carbon atom less than its parent fatty acid ( $C_{n-1}$ ), and this hydrocarbon forms as a result of the loss of the carboxyl group. The other has two carbon atoms less than its parent fatty acid and also forms a double bond at the C1 position ( $C_{n-2:1}$ ) (*17*). Concentrations of hydrocarbons linearly increased with irradiation dose (**Tables 2–5**).

#### **Table 2.** Hydrocarbons Detected in Irradiated Sesame ( $\mu$ g/g of Dried Fat)

hydrocarbons	0	0.5	1	2	4	regression equation and coefficient <sup>a</sup>
1-tetradecene (C <sub>14:1</sub> )	$0.28\pm0.12^b$	$0.77\pm0.53$	$\textbf{0.98} \pm \textbf{0.06}$	$\textbf{2.45}\pm\textbf{0.12}$	$\textbf{2.32}\pm\textbf{0.06}$	$y = 0.535x + 0.558$ $R^2 = 0.9028$
pentadecane (C <sub>15:0</sub> )	$0.31\pm0.05$	$1.16\pm0.10$	$1.51\pm0.35$	$1.82\pm0.06$	$\textbf{2.82} \pm \textbf{0.20}$	y = 0.558x + 0.687 $R^2 = 0.9229$
1,7-hexadecadiene (C <sub>16:2</sub> )		$0.49\pm0.05$	$\textbf{0.76} \pm \textbf{0.03}$	$1.45\pm0.08$	$1.88\pm0.08$	y = 2.344x - 0.809 $R^2 = 0.9254$
1-hexadecene (C <sub>16:1</sub> )		$0.17\pm$ .01	$\textbf{0.32}\pm\textbf{0.04}$	$\textbf{0.38} \pm \textbf{0.01}$	$0.60\pm0.00$	$y = 0.113x + 0.157$ $R^2 = 0.9531$
8-heptadecene (C <sub>17:1</sub> )		$0.24\pm0.01$	$\textbf{0.58} \pm \textbf{0.02}$	$\textbf{0.96} \pm \textbf{0.02}$	$2.05\pm0.09$	$y = 0.506x + 0.008$ $R^2 = 0.9953$
heptadecane (C <sub>17:0</sub> )	$\textbf{0.75}\pm\textbf{0.12}$	$\textbf{0.82}\pm\textbf{0.09}$	$1.26\pm0.05$	$1.51\pm0.01$	$1.86\pm0.32$	y = 0.283x + 0.816 R <sup>2</sup> = 0.9161

<sup>*a*</sup> *x*, irradiation dose (kGy); *y*, hydrocarbon. <sup>*b*</sup> Mean  $\pm$  sd (*n* = 3).

Table 3. Hydrocarbons	s Detected in Irradiat	ed Sesame after	Steaming for 20	min in a Boiling	Water Bath ( <i>i</i>	ug/g of Dried Fat)
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hydrocarbons	0	0.5	1	2	4	regression equation and coefficient <sup>a</sup>
1-tetradecene (C <sub>14:1</sub> )	$0.53\pm0.20^{b}$	$\textbf{0.68} \pm \textbf{0.37}$	$1.13\pm0.05$	$1.20\pm0.66$	$1.54\pm0.17$	y = 0.254x + 0.254
pentadecane (C <sub>15:0</sub> )	$\textbf{0.31} \pm \textbf{0.13}$	$\textbf{0.52}\pm\textbf{0.24}$	$\textbf{0.82}\pm\textbf{0.05}$	$\textbf{0.87} \pm \textbf{0.55}$	$1.70\pm0.71$	$R^2 = 0.9622$ y = 0.198x + 0.135 $R^2 = 0.9415$
1,7-hexadecadiene (C <sub>16:2</sub> )		$1.02\pm0.81$	$1.77\pm0.11$	$1.96\pm0.55$	$\textbf{2.63} \pm \textbf{0.06}$	y = 0.620x - 0.384
1-hexadecene (C <sub>16:1</sub> )	$\textbf{0.12}\pm\textbf{0.03}$	$\textbf{0.25}\pm\textbf{0.12}$	$\textbf{0.63} \pm \textbf{0.25}$	$0.91\pm0.58$	$1.69\pm0.61$	$R^2 = 0.9517$ y = 0.380x - 0.420 $R^2 = 0.9221$
8-heptadecene (C <sub>17:1</sub> )		$\textbf{0.49} \pm \textbf{0.40}$	$1.14\pm0.08$	$1.50\pm1.03$	$2.55 \pm 0.15$	y = 0.611x - 0.697
heptadecane (C <sub>17:0</sub> )	$0.47\pm0.23$	$\textbf{0.67} \pm \textbf{0.24}$	$\textbf{0.76} \pm \textbf{0.05}$	$\textbf{0.84} \pm \textbf{0.04}$	$1.00\pm0.27$	$R^{2} = 0.9723$ y = 0.123x + 0.379 $R^{2} = 0.9731$

<sup>*a*</sup> *x*, irradiation dose (kGy); y, hydrocarbon. <sup>*b*</sup> Mean  $\pm$  sd (n = 3).

Table 4. I	-lydrocarbons	Detected in	Irradiated	Sesame a	fter Roasting	g at 220	°C for	10 min	$(\mu q/q o$	f Dried Fa	t)
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hydrocarbons	0	0.5	1	2	4	regression equation and coefficient <sup>a</sup>
1-tetradecene (C <sub>14:1</sub> )	$0.39\pm0.05^{b}$	$0.51\pm0.07$	$\textbf{0.54} \pm \textbf{0.40}$	$\textbf{0.64} \pm \textbf{0.02}$	$\textbf{0.93} \pm \textbf{0.27}$	$y = 0.121x + 0.239$ $R^2 = 0.8805$
pentadecane (C <sub>15:0</sub> )	$\textbf{0.56} \pm \textbf{0.06}$	$\textbf{0.78} \pm \textbf{0.05}$	$1.10\pm0.35$	$1.56\pm0.87$	$1.82 \pm 1.21$	y = 0.330x + 0.174 $R^2 = 0.9868$
1,7-hexadecadiene ( $C_{16:2}$ )		$\textbf{0.58} \pm \textbf{0.05}$	$\textbf{0.60} \pm \textbf{0.12}$	$1.53\pm0.06$	$\textbf{2.27} \pm \textbf{0.01}$	$y = 0.549x - 0.651$ $R^2 = 0.9331$
1-hexadecene (C <sub>16:1</sub> )	$\textbf{0.16} \pm \textbf{0.03}$	$\textbf{0.21}\pm\textbf{0.01}$	$\textbf{0.28}\pm\textbf{0.01}$	$\textbf{0.39} \pm \textbf{0.44}$	$\textbf{0.65} \pm \textbf{0.39}$	y = 0.116x - 0.010 $R^2 = 0.8883$
8-heptadecene (C <sub>17:1</sub> )		$\textbf{0.31} \pm \textbf{0.13}$	$\textbf{0.32}\pm\textbf{0.05}$	$\textbf{0.96} \pm \textbf{0.01}$	$1.87\pm0.21$	$y = 0.439x - 0.625$ $R^2 = 0.8671$
heptadecane (C <sub>17:0</sub> )	$\textbf{0.56} \pm \textbf{0.02}$	$0.57\pm0.01$	$\textbf{0.76} \pm \textbf{0.12}$	$1.19\pm0.80$	$1.45\pm0.85$	y = 0.240x + 0.186 $R^2 = 0.9135$

<sup>*a*</sup> *x*, irradiation dose (kGy); *y*, hydrocarbon. <sup>*b*</sup> Mean  $\pm$  sd (*n* = 3).

They, however, were detected in different concentrations at the same dose, which were mainly due to the composition of fatty acids in different foods, such as soybean (18), peanut (19), and cashew nut (20). Hydrocarbons, such as 1-tetradecene ( $C_{14:1}$ ), pentadecane ( $C_{15:0}$ ), and heptadecane ( $C_{17:0}$ ), were found in non-irradiated samples. These results were similar to those reported by Choi and Hwang (21), in which pentadecane ( $C_{15:0}$ ) and heptadecane ( $C_{15:0}$ ) and heptadecane ( $C_{15:0}$ ) and heptadecane ( $C_{16:2}$ ), in which pentadecane ( $C_{16:2}$ ), 1-hexadecene ( $C_{16:1}$ ), and 8-heptadecene ( $C_{17:1}$ ), were only detected in irradiated samples (**Table 2**). Concentrations of radiation-induced hydrocarbons increased with irradiation dose, and they were detectable even at 0.5 kGy. The correlation coefficients obtained between irradiation dose and concentration of hydro-

carbons ranged from 0.9028 to 0.9953. The three types of treatments, such as steaming, roasting, and oil extraction after roasting by the commercial pressing method, affected the detection levels of hydrocarbons compared to nontreated samples (**Tables 2**-5). However, the non-irradiated sesame seeds after treatments showed a different pattern from the nontreated samples. Among the non-irradiated samples, the hydrocarbon 1-hexadecene (C<sub>16:1</sub>) was present only in treated samples. The hydrocarbons, 1,7-hexadecadiene (C<sub>16:2</sub>) and 8-heptadecene (C<sub>17:</sub> 1), were detected only in the irradiated samples before and after treatments at more than 0.5 kGy. Thus, these two hydrocarbons could be used as markers to identify irradiated sesame seeds. The amount of these hydrocarbons increased almost linearly ( $R^2 = 0.8671-0.9953$ ) with the applied doses (**Tables 2**-5).

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hydrocarbons	0	0.5	1	2	4	regression equation and coefficient <sup>a</sup>
1-tetradecene (C <sub>14:1</sub> )	$0.47\pm0.08^{b}$	$\textbf{0.55} \pm \textbf{0.37}$	$\textbf{0.62}\pm\textbf{0.38}$	$\textbf{0.72}\pm\textbf{0.09}$	$1.12\pm0.07$	$y = 0.147x + 0.255$ $R^2 = 0.8359$
pentadecane (C <sub>15:0</sub> )	$\textbf{0.60} \pm \textbf{0.23}$	$\textbf{0.80} \pm \textbf{0.31}$	$\textbf{0.89} \pm \textbf{0.60}$	$1.26\pm0.88$	$1.28\pm0.09$	$y = 0.182x + 0.420$ $R^2 = 0.9402$
1,7-hexadecadiene (C <sub>16:2</sub> )		$\textbf{0.54} \pm \textbf{0.01}$	$\textbf{0.87} \pm \textbf{0.02}$	$1.71\pm0.07$	$3.50\pm0.20$	y = 0.817x - 1.127 $R^2 = 0.8950$
1-hexadecene (C <sub>16:1</sub> )	$\textbf{0.40} \pm \textbf{0.26}$	$\textbf{0.56} \pm \textbf{0.50}$	$\textbf{0.71} \pm \textbf{0.59}$	$0.86\pm0.09$	$1.70\pm0.28$	y = 0.290x - 0.024 $R^2 = 0.8175$
8-heptadecene (C <sub>17:1</sub> )		$\textbf{0.14} \pm \textbf{0.20}$	$\textbf{0.45} \pm \textbf{0.04}$	$1.54 \pm 1.00$	$2.32\pm0.01$	$y = 0.604x + 0.922$ $R^2 = 0.9085$
heptadecane (C <sub>17:0</sub> )	$\textbf{0.36} \pm \textbf{0.17}$	$\textbf{0.43} \pm \textbf{0.04}$	$\textbf{0.52}\pm\textbf{0.03}$	$0.71\pm0.25$	$\textbf{0.82}\pm\textbf{0.08}$	$y = 0.120x + 0.208$ $R^2 = 0.9711$

<sup>a</sup> x, irradiation dose (kGy); y, hydrocarbon. <sup>b</sup> Mean  $\pm$  sd (n = 3).

Table 6.	2-Alkvlcvclobuta	Inones Detected	in Irradiated	Sesame after	Steaming.	Roasting, an	d Oil Extraction	(µa/a of [	Dried Fa	t)
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sample	2-akylcyclobutanones	0	0.5	1	2	4	regression equation and coefficient <sup>a</sup>
	2-dodecylcyclobutanone (DCB, C <sub>12:0</sub> )		$0.21\pm0.05^{\textit{b}}$	$0.47\pm0.07$	$0.53\pm0.05$	$0.69\pm0.50$	$y = 0.115x + 0.258$ $R^2 = 0.8069$
raw sesame	2-tetradecylcyclobutanone (2-TCB, C <sub>14:0</sub> )		$\textbf{0.18} \pm \textbf{0.01}$	$0.23\pm0.00$	$\textbf{0.33}\pm\textbf{0.07}$	$0.42\pm0.06$	y = 0.068x + 0.164 $R^2 = 0.9569$
	2-(5'-tetradecenyl) cyclobutanone (2-TeCB, C <sub>14:1</sub> )		$\textbf{0.36} \pm \textbf{0.00}$	$\textbf{0.42}\pm\textbf{0.12}$	$\textbf{0.48} \pm \textbf{0.06}$	$0.97\pm0.18$	y = 0.175x + 0.229 $R^2 = 0.9418$
	2-dodecylcyclobutanone (DCB, C <sub>12:0</sub> )		$\textbf{0.20}\pm\textbf{0.03}$	$0.48\pm0.06$	$0.57\pm0.04$	$0.69\pm0.05$	y = 0.1172x + 0.265 $R^2 = 0.7567$
steamed sesame	2-tetradecylcyclobutanone (2-TCB, C <sub>14:0</sub> )		$\textbf{0.21}\pm\textbf{0.02}$	$0.24\pm0.02$	$\textbf{0.33}\pm\textbf{0.08}$	$0.44\pm0.05$	y = 0.066x + 0.180 $R^2 = 0.9882$
	2-(5'-tetradecenyl) cyclobutanone (2-TeCB, C <sub>14:1</sub> )		$\textbf{0.38} \pm \textbf{0.04}$	$\textbf{0.43} \pm \textbf{0.13}$	$\textbf{0.49} \pm \textbf{0.05}$	$0.95\pm0.18$	y = 0.164x + 0.254 $R^2 = 0.9412$
	2-dodecylcyclobutanone (DCB, C <sub>12:0</sub> )		$\textbf{0.26} \pm \textbf{0.24}$	$0.50\pm0.60$	$\textbf{0.64} \pm \textbf{2.43}$	$\textbf{0.70} \pm \textbf{2.31}$	y = 0.108x + 0.324 $R^2 = 0.7239$
roasted sesame	2-tetradecylcyclobutanone (2-TCB, C <sub>14:0</sub> )		$0.21\pm0.02$	$\textbf{0.25}\pm\textbf{0.04}$	$0.41\pm0.07$	$0.46\pm0.11$	y = 0.073x + 0.197 $R^2 = 0.8577$
	2-(5'-tetradecenyl) cyclobutanone (2-TeCB, C <sub>14:1</sub> )		$\textbf{0.39} \pm \textbf{0.05}$	$\textbf{0.48} \pm \textbf{0.20}$	$0.56\pm0.05$	$1.03\pm0.05$	y = 0.181x + 0.275 $R^2 = 0.9670$
roasted oil	2-dodecylcyclobutanone (DCB, C <sub>12:0</sub> )		$\textbf{0.24}\pm\textbf{0.04}$	$0.32\pm0.02$	$0.57\pm0.02$	$0.65\pm0.03$	y = 0.117x + 0.225 $R^2 = 0.8565$
	2-tetradecylcyclobutanone (2-TCB, C <sub>14:0</sub> )		$\textbf{0.23}\pm\textbf{0.01}$	$\textbf{0.30}\pm\textbf{0.01}$	$0.50\pm0.05$	$\textbf{0.59}\pm\textbf{0.04}$	$y = 0.103x + 0.213$ $B^2 = 0.8913$
	2-(5'-tetradecenyl) cyclobutanone (2-TeCB, C <sub>14:1</sub> )		$0.37\pm0.02$	$\textbf{0.54} \pm \textbf{0.08}$	$0.67\pm0.10$	$1.05\pm0.07$	$y = 0.185x + 0.310$ $R^2 = 0.9873$

<sup>*a*</sup> *x*, irradiation dose (kGy); *y*, 2-alkylcyclobutanone. <sup>*b*</sup> Mean  $\pm$  sd (*n* = 3).

2-Alkylcyclobutanone Characteristics. The 2-alkylcyclobutanones are ordinarily formed by radiolysis of triglycerides in foods treated by accelerated electron beams, X-rays, or  $\gamma$ radiation. These cyclic compounds, with a ring of four carbon atoms, have the same carbon number as the parent fatty acid (22). Particularly, 2-alkylcyclobutanone was detected entirely in irradiated foods. Furthermore, there has never been a report that these compounds were found in non-irradiated foods treated by other food processes, such as freezing, heating, microwave heating, UV irradiation, high-pressure processing, or simple preservation treatment (23, 24). The major fatty acids in irradiated sesame seeds can be formed into four 2-alkylcyclobutanones, such as 2-dodecylcyclobutanone (DCB) from palmitic acid, 2-tetradecylcyclobutanone (2-TCB) from stearic acid, 2-(5'tetradecenyl)cyclobutanone (2-TeCB) from oleic acid, and 2-(5',8'-tetradecadienyl)cyclobutanone from linoleic acid. The presence of 2-(5',8'-tetradecadienyl)cyclobutanone could not be confirmed in this study because this standard was not purchased. The concentrations of the three detected 2-alkylcyclobutanones linearly increased with the irradiation dose. These compounds could be detected at doses  $\geq 0.5$  kGy but not in non-irradiated samples. Generally, the amount of these compounds increased almost linearly ( $R^2 = 0.7239 - 0.9882$ ) with the applied doses (**Table 6**). In particular, the increase in 2-TeCB is highest ( $R^2 = 0.9412 - 0.9873$ ), which may be due to the abundance of oleic acid (38.7%) in sesame seeds. The three types of treatment had no significant effect on the levels of 2-alkylcyclobutanones.

In conclusion, hydrocarbons and 2-alkylcyclobutanones formed during the irradiation of sesame seeds increased with the irradiation dose. The hydrocarbons, 1,7-hexadecadiene and 8-heptadecene, could be used as markers to identify irradiated sesame seeds. 2-Alkylcyclobutanones were detected only in the treated irradiated samples at doses  $\geq 0.5$  kGy, which could also be used as markers to identify irradiated sesame seeds. One of the 2-alkylcyclobutanones, 2-TeCB, was found to have the highest concentration in sesame seeds.

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