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Analysis of Radiation-Induced Hydrocarbons and 2-Alkylcyclobutanones from Dried Shrimps (*Penaeus aztecus*)

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ABSTRACT

We investigated the usefulness of hydrocarbons and 2-alkylcyclobutanones as markers for irradiated, dried shrimps. A method was developed to detect the irradiation of dried shrimps (*Penaeus aztecus*) by identifying radiation-induced hydrocarbons and 2-alkylcyclobutanones extracted from dried shrimps, which were separated by Florisil column chromatography and identified by a method using gas chromatography/mass spectrometry. Radiation-induced hydrocarbons and 2-alkylcyclobutanones are formed from the fatty acids of the irradiated fats. The quantity of the hydrocarbons and 2-alkylcyclobutanones formed from some fatty acids is related to the composition of fatty acids in a sample. The concentrations of hydrocarbons and 2-alkylcyclobutanones increased with radiation dose. The major hydrocarbons in the irradiated, dried shrimps were 1-tetradecene and pentadecane from palmitic acid; small amounts of heptadecane and 1-hexadecene from stearic acid; and 8-heptadecene and 1,7-hexadecadiene from oleic acid. 2-(5′-Tetradecenyl)cyclobutanone, one of the 2-alkylcyclobutanones, was present at the highest concentration. The radiation-induced hydrocarbons and 2-alkylcyclobutanones were detected at radiation doses of 0.5 kGy and greater. These compounds were not detected in nonirradiated, dried shrimps.

The irradiation of food has been recognized for many years as a means to reduce food losses, improve food safety, and extend shelf life. Furthermore, irradiation can be an effective way of reducing the incidence of foodborne disease and treating a variety of potential problems in food supplies. The treatment of food with ionizing radiation is one of the most thoroughly researched techniques available to the food processing industry, and its use is currently permitted in 52 countries for the treatment of approximately 250 food products *(7).* In 1981, the JECFI (FAO/IAEA/WHO Expert Committee on the Wholesomeness of Irradiated Food) stated that "the irradiation of any food commodity up to an overall average dose of 10 kGy presents no toxicological hazard, introduces no special nutritional or microbiological problems" *(24).* It has recently been reported that "food irradiated to any dose appropriate to achieve the intended technological objective is both safe to consume and nutritionally adequate" *(25).* Although properly irradiated food is safe and wholesome, consumers should be able to make a choice between irradiated and nonirradiated products. To this end, labeling is indispensable. To check for compliance with existing regulations, suitable methods must be available for the reliable authentication of irradiated foods. Some research on detection methods has been carried out during the past decades, but a significant move toward more research arose because of a joint FAO/IAEA worldwide program on the Analytical Detection Methods for the Irradiation Treatment of Foods *(13)* and a European program by the Community Bureau of Reference *(18).* An important landmark in the research work came in December 1996, when the European Committee for Standardization adopted five methods as European Standards *(1).* These methods include electron spin resonance spectroscopy methods for foods containing bone and crystalline cellulose and thermoluminescence for the detection of foods with extractable silicate minerals and the detection of hydrocarbons and 2-alkylcyclobutanones produced by the irradiation of foods containing fats.

Dubravcic and Nawar *(6)* reported that hydrocarbons are formed from the fatty acids in irradiated foods that contain fat and that the type of hydrocarbons formed have either one or two fewer carbon atoms (C_{n-1}) or (C_{n-2}), respectively, than their parent fatty acids. All 2-alkylcyclobutanones have the same number of carbon atoms as their parent fatty acids and are substituted with an alkyl group located in the second ring position. LeTellier and Nawar *(11)* first identified 2-alkylcyclobutanones as radiolytic products from pure triglycerides irradiated with 60 kGy. Since then, a number of studies have been performed on their use as detection markers for irradiated foods *(2, 3, 5, 13, 14, 19, 21–23),* but the systematic data developed to date have been insufficient to apply to every food.

An increased demand for marine products, brought about by growth in the national income as well as a preference for these products, has expanded importation because of the lack of a local market. Of these products, a large amount of dried shrimps are imported because of con-

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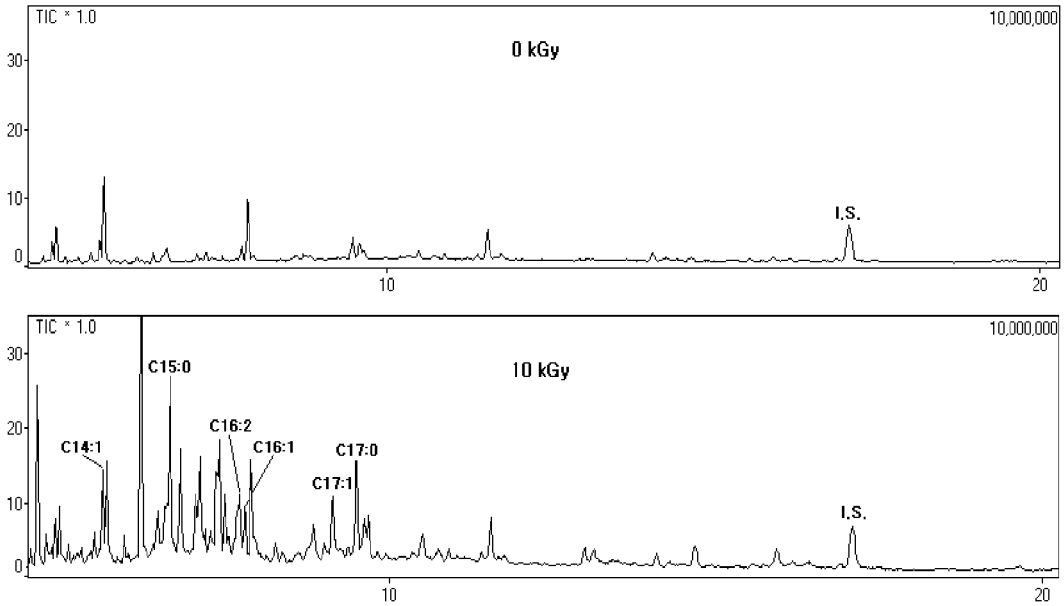

FIGURE 1. *Chromatograms for the hydrocarbons from nonirradiated and 10-kGy–irradiated, dried shrimps.*

sumer preference and ease of cooking. Therefore, this study was performed to identify the hydrocarbons and 2-alkylcyclobutanones formed during the irradiation of dried shrimps (*Penaeus aztecus*), to provide a quantitative method for the identification of irradiated foods, and, furthermore, to enhance consumer confidence by labeling the product with the results of the analysis.

MATERIALS AND METHODS

Materials. The dried shrimps (*P. aztecus*) were purchased from a local supermarket and irradiated with doses of 0.5, 1, 3, 5, and 10 kGy at 12 ± 1°C using a ^{60}Co γ-irradiator at the Korea Atomic Energy Research Institute (Daejeon, Korea). The dose rate was 2.5 kGy/h, with a dose rate error of ±0.02 kGy. Absorbed doses were monitored with either a radical or a ceric-cerous dosimeter. The irradiated samples and nonirradiated control were stored at −18°C until required for the experiments.

Reagents. The standard hydrocarbons and 2-alkylcyclobutanones were purchased from TeLA Company (Berlin, Germany). High-performance liquid chromatography grade solvents (*n*-hexane and diethyl ether) were purchased from Fisher Scientific (Pittsburgh, Pa.) and further distilled with spiral-packed double-distilling apparatus prior to use. The Florisil (60- to 100-mesh, Fisher) was heated overnight at 550°C to remove possible contaminants. Prior to use, the Florisil was heated for at least 5 h at 130°C in a dry oven and then cooled in a desiccator. Afterward, 3% water (wt/wt) was added to separate the hydrocarbons, or 20% water (wt/wt) was added to separate the 2-alkylcyclobutanones, and the mixture was shaken for at least 20 min. These mixtures were stored at room temperature for 10 to 12 h. Florisil deactivated in this way was used for 3 days. After this time, the Florisil was reheated to 130°C and deactivated again.

Extraction of fat from dried shrimps. A Soxtec HT2 (Foss, France) apparatus was used to extract the fat from the sample. Five grams each of homogenized dried shrimp and anhydrous sodium sulfate were mixed; the mixture was weighed into an extraction thimble, and the thimble was inserted into the Soxtec apparatus. Approximately 50 ml of *n*-hexane was added to a preweighed extraction cup and inserted into the extraction unit. The soluble lipids were extracted automatically into the solvent in two stages (rinsing and boiling), and the solvent was then recovered. The sample was boiled for 60 min at 115°C and rinsed for 15 min. The extracted fat was placed in N_2-filled vials and stored at −20°C.

Separation of hydrocarbons. Separation of hydrocarbons was performed on Florisil resin, according to the method of Kim et al. *(8).* Twenty-five grams of deactivated Florisil was packed into a glass column (200 by 20 mm). Anhydrous sodium sulfate was added to the top of the column in a 1-cm layer. One gram of extracted fat was mixed with an international standard (1 ml of

TABLE 1. *Concentrations of radiation-induced hydrocarbons in dried shrimps (μg/g fat)*

Irradiation dose (kGy)	Palmitic acid		Stearic acid		Oleic acid	
	$C_{15:0}$	$C_{14:1}$	$C_{17:0}$	$C_{16:1}$	$C_{17:1}$	$C_{16:2}$
0	—	—	—	—	—	—
0.5	2.093 (±0.10)[a]	1.792 (±0.11)	2.261 (±0.17)	0.510 (±0.08)	0.470 (±0.14)	0.408 (±0.11)
1	4.665 (±0.39)	2.670 (±0.06)	2.969 (±0.36)	0.846 (±0.06)	1.248 (±0.23)	0.968 (±0.05)
3	7.529 (±1.05)	3.985 (±0.22)	3.218 (±0.87)	2.378 (±0.19)	3.066 (±0.22)	2.187 (±0.16)
5	8.857 (±1.02)	4.794 (±0.20)	4.603 (±0.87)	2.748 (±0.18)	4.415 (±0.30)	3.732 (±0.23)
10	16.258 (±0.60)	7.198 (±0.78)	11.797 (±1.02)	5.476 (±0.64)	7.444 (±0.13)	5.138 (±0.45)

[a] Mean (±standard deviation); $n = 3$.

n-eicosane [4 μg/ml in hexane]), applied to the column, and eluted with 60 ml of *n*-hexane at a flow rate of 3 ml/min. The hexane eluent was concentrated to a volume of 2 ml in a rotary vacuum evaporator and further concentrated to a volume of 0.5 ml using nitrogen.

Separation of 2-alkylcyclobutanones. The Florisil column was prepared as previously described. The extracted fat (0.2 g) was mixed with 1 ml of 2-cyclohexylcyclohexanone (1 μg/ml in hexane) as the international standard, applied to the column, and eluted, first with 150 ml of *n*-hexane and then with 120 ml of diethyl ether–hexane (2:98 [vol/vol]), at a flow rate of 3 ml/min. The latter hexane fraction was concentrated to a volume of 2 ml in a rotary vacuum evaporator and further concentrated to a volume of 0.2 ml using nitrogen.

Gas chromatography/mass spectrometry analysis of hydrocarbons. The gas chromatography/mass spectrometry (GC/MS) analyses were carried out on a Shimadzu GC/MS QP-5050 spectrometer (Kyoto, Japan) in electron impact mode using a DB-5 column (inside diameter, 30 m by 0.32 mm; 0.25-μm film thickness; J & W Scientific, Folsom, Calif.). The ionization voltage was set at 70 eV, and the injector and ion source temperatures were kept at 250°C. The oven temperature was programmed as follows: (i) from 60 to 170°C with a ramp rate of 25°C/min, (ii) to 205°C with a ramp rate of 2°C/min, and (iii) to 270°C with a ramp rate of 10°C/min. Helium was used as the carrier gas, at a flow rate of 1.0 ml/min. One microliter of the sample was injected in the splitless mode for 2 min and then in a split mode (20:1). The hydrocarbons were identified by comparing the retention time and mass spectrum of peaks, as shown in the total ion chromatogram, with those of an authentic hydrocarbon standard. The concentration of each hydrocarbon in the fat was determined using *n*-eicosane (4 μg/ml) as an internal standard.

GC/MS analysis of 2-alkylcyclobutanones. The GC/MS analyses were carried out on a Shimadzu GC/MS QP-5050 spectrometer in electron impact mode using a DB-5 column (inside diameter, 30 m by 0.32 mm; 0.25-μm film thickness; J & W Scientific). The injector and ion source temperatures were kept at 250 and 290°C, respectively. The oven temperature was programmed as follows: (i) from 120°C (1 min) to 160°C with a ramp rate of 15°C/min, (ii) to 175°C with a ramp rate of 0.5°C/min, and (iii) to 290°C with a ramp rate of 30°C/min (10 min). Two microliters of the sample was injected in the splitless mode for 1 min and was then injected in a split mode (20:1). The other conditions were the same as for the hydrocarbons.

The 2-alkylcyclobutanones were analyzed by GC/MS using the SIM (selected ion monitoring mode). The SIM for the 2-alkylcyclobutanones was set for 2-dodecylcyclobutanone and 2-tetradecylcyclobutanone using the *m/z* 98 and *m/z* 112 ions, and the SIM for 2-(5′-tetradecenyl)cyclobutanone was set using the *m/z* 67, *m/z* 81, *m/z* 98, and *m/z* 109 ions. The peaks produced and the retention times and ion ratios were compared with those of the 2-alkylcyclobutanone standards. Mass spectra of the 2-alkylcyclobutanones were confirmed by GC/MS in the full-scan mode. The concentration of each 2-alkylcyclobutanone in the fat was determined using 2-cyclohexylcyclohexanone (1 μg/ml) as an internal standard.

RESULTS AND DISCUSSION

Radiation-induced hydrocarbons from irradiated, dried shrimps. The formation of lipid-derived long-chain hydrocarbons was based on the observation that, when fatty acids are exposed to high-energy radiation, they undergo

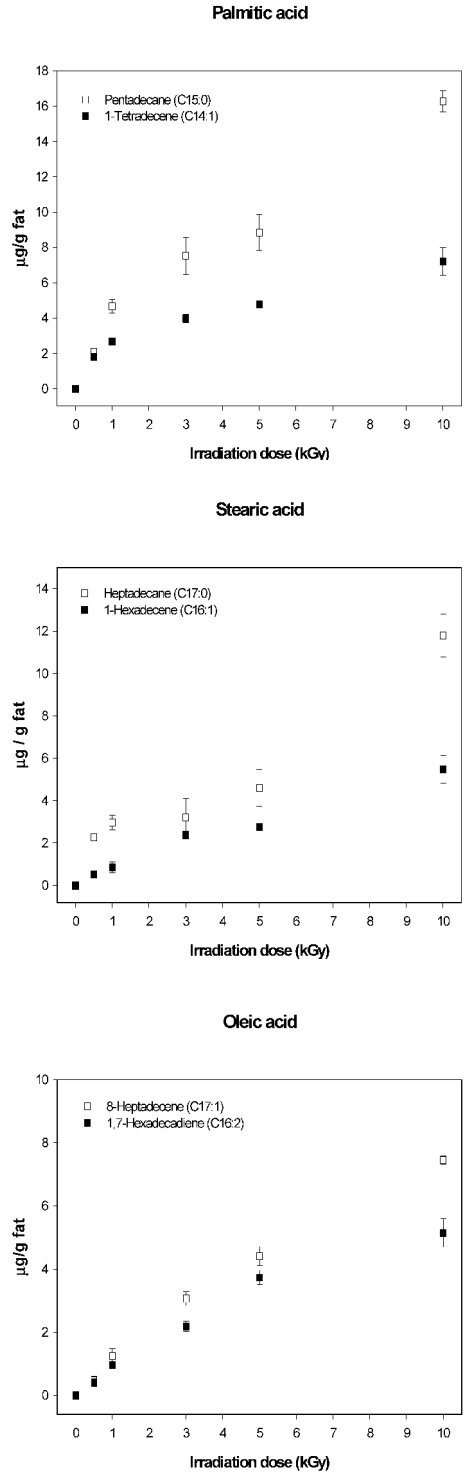

FIGURE 2. *Irradiation dose dependence of radiation-induced hydrocarbons in dried shrimps.*

preferential cleavage in the ester carbonyl region, giving rise to certain key compounds in relatively large amounts. The two hydrocarbons of most interest that are formed are those with one or two carbon atoms fewer than their parent fatty acids (C_{n-1}) or ($C_{n-2:1}$), respectively, and an additional double bond in the first position *(7, 20).* From this information, we can deduce that pentadecane ($C_{15:0}$) and 1-tetradecene ($C_{14:1}$) (from palmitic acid), heptadecane ($C_{17:0}$) and 1-hexadecene ($C_{16:1}$) (from stearic acid), 8-hep-

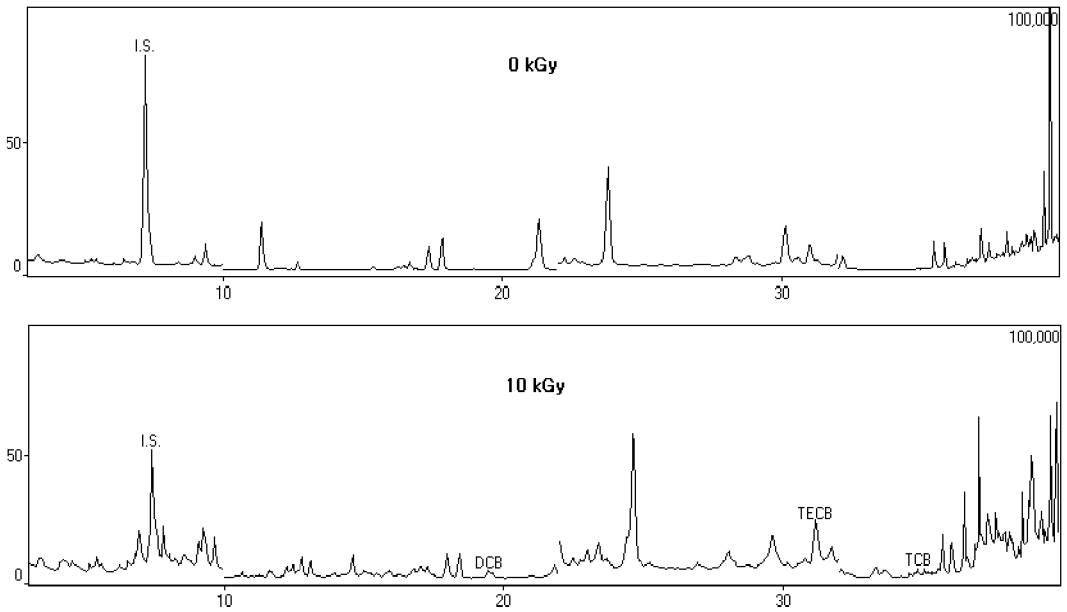

FIGURE 3. *Chromatograms for the 2-alkylcyclobutanones from nonirradiated and 10-kGy–irradiated, dried shrimps. DCB indicates 2-dodecylcyclobutanone; TECB, 2-(5′-tetradecenyl)cyclobutanone; and TCB, 2-tetradecylcyclobutanone.*

tadecene ($C_{17:1}$) and 1,7-hexadecadiene ($C_{16:2}$) (from oleic acid), and 6,9-heptadecadiene ($C_{17:2}$) and 1,7,10-hexadecatriene ($C_{16:3}$) (from linoleic acid) were formed.

The major fatty acids in the shrimps were palmitic acid (10.3 to 17.4%), eicosapetaenoic acid (10.6 to 15.8%), docosahexaenoic acid (7.75 to 12.13%), oleic acid (5.6 to 7.8%), and stearic acid (6.1 to 6.9%) *(4).* According to the fatty acid composition, the major hydrocarbons were presupposed to be from palmitic acid, eicosapetaenoic acid, and docosahexaenoic acid. However, the radiation-induced hydrocarbons from eicosapetaenoic acid and docosahexaenoic acid cannot be confirmed, because there are no hydrocarbon standards for them. Therefore, the radiolytic hydrocarbons of palmitic, stearic, and oleic acids were determined, and the amounts formed were compared.

Figure 1 shows the gas chromatograms of the radiation-induced hydrocarbons for the nonirradiated shrimps and the dried shrimps irradiated with 10 kGy. The results were in agreement with the irradiation lipid degradation patterns proposed by Nawar *(16),* and a number of radiolytic hydrocarbons could be detected, with concentrations of the hydrocarbons increasing with irradiation dose. The data of the quantitative analysis for the radiation-induced hydrocarbons from the shrimp samples are shown in Table 1. Also, as indicated in Figure 2, hydrocarbons were detected at different concentrations for the same irradiation dose, depending on the composition of the fatty acids in the shrimps. This has previously been noted with pork, beef, and chicken *(20),* shrimps and chicken *(15),* and various other foods *(9–11, 17).*

Pentadecane and 1-tetradecene are the two hydrocarbons that were formed from the irradiation of palmitic acid. Pentadecane was detected in relatively large amounts compared to 1-tetradecene at all irradiation doses, with a mean pentadecane/1-tetradecene ratio of 1.78. Also, these hydrocarbons were present in higher concentrations than were the other radiolytic hydrocarbons because of the composition of the fatty acids. The linear regression coefficients (r^2) of pentadecane and 1-tetradecene were 0.957 and 0.899, respectively, and the concentrations increased linearly with radiation dose. Heptadecane and 1-hexadecene were formed from the irradiation of stearic acid, and they were detected by our GC/MS procedure. Their concentrations correlated linearly with increased radiation doses. The mean heptadecane/1-hexadecene ratio was 2.24; the $C_{n-1}/C_{n-2:1}$ ratio was higher than for the other hydrocarbon ratios, such as the pentadecane/1-tetradecene and 8-heptadecene/1,7-hexadecadiene ratios. The linear regression coefficients (r^2) for heptadecane and 1-hexadecene were 0.932 and 0.981, respectively. 8-Heptadecene and 1,7-hexadecadiene, from oleic acid, were detected with the same radiolysis patterns with respect to the observation that the C_{n-1} hydrocarbons were formed in higher concentrations than the $C_{n-2:1}$ hydrocarbon. The mean $C_{n-1}/C_{n-2:1}$ ratio was 1.29. Similar to other hydrocarbons, the concentrations of 8-heptadecene and 1,7-hexadecadiene in the irradiated, dried shrimps in-

TABLE 2. *Concentration of radiation-induced 2-alkylcyclobutanones in dried shrimps (μg/g fat)*

Irradiation dose (kGy)	2-Dodecylcyclobutanone (DCB)	2-Tetradecylcyclobutanone (TCB)	2-(5′-Tetradecenyl)cyclobutanone (TECB)
0	—	—	—
0.5	0.014 (±0.001)[a]	0.006 (±0.001)	0.565 (±0.06)
1	0.036 (±0.001)	0.0159 (±0.006)	0.853 (±0.19)
3	0.070 (±0.001)	0.034 (±0.02)	1.433 (±0.08)
5	0.120 (±0.03)	0.058 (±0.009)	2.314 (±0.32)
10	0.351 (±0.01)	0.130 (±0.008)	3.012 (±0.30)

[a] Mean (±standard deviation); $n = 3$.

creased linearly with the irradiation dose, with linear regression coefficients (r^2) of 0.983 and 0.950, respectively.

The major hydrocarbons formed, based on the composition of fatty acids and their degradation mechanism in dried shrimps, were pentadecane, 1-tetradecene, heptadecane, and 1-hexadecene; C_{n-1} hydrocarbons, such as pentadecane and heptadecane, were detected in higher concentrations than $C_{n-2:1}$ hydrocarbons, such as 1-tetradecene and 1-hexadecene. Therefore, these hydrocarbons could be detected whether the sample had been irradiated or not. Using 8-heptadecene and 1,7-hexadecadiene as irradiation markers somewhat detracted from the value of this technique because of their relatively low concentrations in irradiated shrimp. Radiation-induced hydrocarbons were detected at doses of 0.5 kGy and greater but were not detected in nonirradiated shrimps.

Radiation-induced 2-alkylcyclobutanones from irradiated, dried shrimps. 2-Alkylcyclobutanones are cyclic compounds that have the same number of carbon atoms as their precursor fatty acids, with the alkyl group in the second ring position. Therefore, when the four major fatty acid precursors found in most lipid-containing foods—namely, palmitic, stearic, oleic, and linoleic acid—are exposed to irradiation, the 2-alkylcyclobutanones—2-dodecylcyclobutanone, 2-tetradecylcyclobutanone, 2-(5′-tetradecenyl)cyclobutanone, and 2-(5′,8′-tetradecadienyl)cyclobutanone, respectively—are formed *(11)*.

Dried shrimps were irradiated at doses of 0.5, 1, 3, 5, and 10 kGy, and nonirradiated shrimps were used as controls. During the irradiation of the dried shrimps, 2-dodecylcyclobutanone, 2-tetradecylcyclobutanone, and 2-(5′-tetradecenyl)cyclobutanone are formed, but the formation of the 2-(5′,8′-tetradecadienyl)cyclobutanone could not be confirmed, because a standard solution is not available.

Figure 3 shows the chromatograms of the 2-alkylcyclobutanones of the nonirradiated shrimps and the 10-kGy–irradiated, dried shrimps. The data of the quantitative analyses for the radiation-induced 2-alkylcyclobutanones in the dried shrimp samples are shown in Table 2. The observation that the concentrations of 2-alkylcyclobutanones increased with radiation dose when used as markers of radiation-induced hydrocarbons was confirmed by LeTellier and Nawar *(11)*.

Of the 2-alkylcyclobutanones, 2-(5′-tetradecenyl)cyclobutanone, formed from oleic acid, had the highest concentration per amount of precursor fatty acid in the shrimps. The concentration of the 2-dodecylcyclobutanone was higher than that of the 2-tetradecylcyclobutanone, because more palmitic acid than stearic acid is present in shrimps. The concentrations of 2-dodecylcyclobutanone and 2-tetradecylcyclobutanone reflected the ratio of their precursor fatty acids but were considerably lower than the concentration of 2-(5′-tetradecenyl)cyclobutanone. This result is similar to the report on irradiation markers in irradiated prawns by McMurray et al. *(12)*.

When the concentrations of radiation-induced 2-alkylcyclobutanones were plotted against the amount of fats, a linear response was observed with the radiation dose (Fig. 4). The linear regression coefficients (r^2) for 2-dodecylcyclobutanone, 2-tetradecylcylobutanone, and 2-(5′-tetradecenyl)cyclobutanone were 0.967, 0.995, and 0.920, respectively. Radiation-induced 2-alkylcyclobutanones (eg, 2-dodecylcyclobutanone, 2-tetradecylcyclobutanone, and 2-(5′-tetradecenyl)cyclobutanone) were detected at doses of 0.5 kGy and greater but were not detected in the nonirradiated samples.

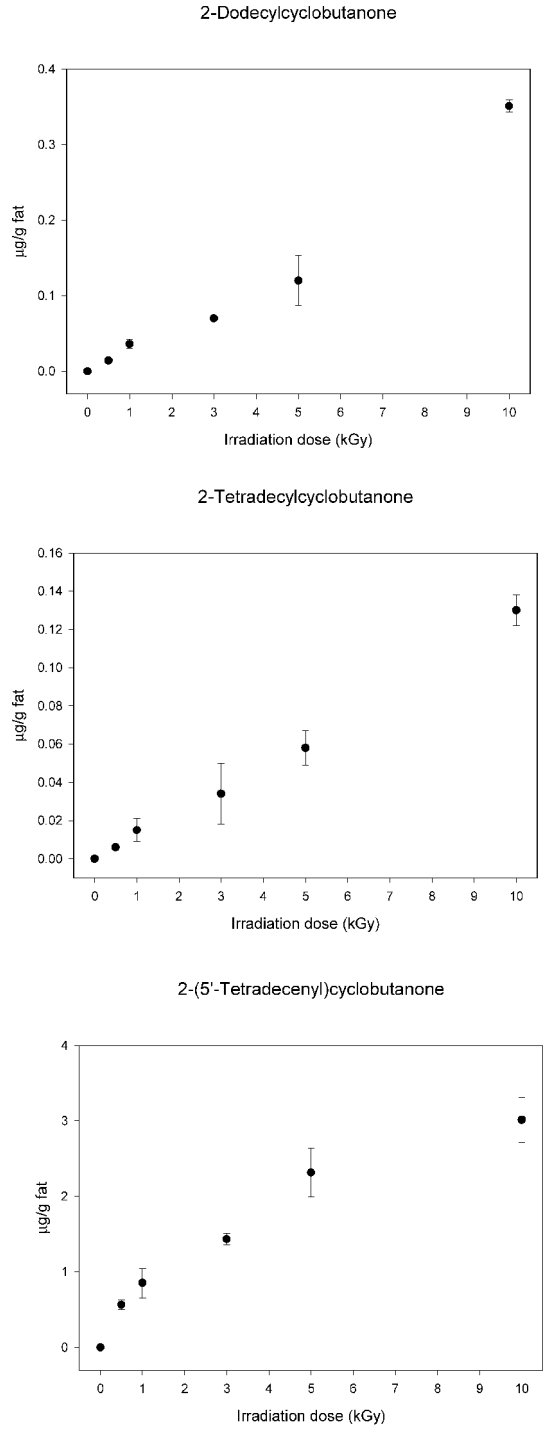

FIGURE 4. *Irradiation dose dependence of radiation-induced 2-alkylcyclobutanones in dried shrimps.*

In conclusion, the hydrocarbons and 2-alkylcyclobutanones formed by the irradiation of dried shrimps in-

creased proportionally to the radiation dose and were detected at doses of 0.5 kGy and greater. The major hydrocarbons detected were pentadecane, 1-tetradecene, heptadecane, and 1-hexadecene. One of the 2-alkylcyclobutanones, 2-(5′-tetradecenyl)cyclobutanone, had the highest concentration in the irradiated, dried shrimps. These major hydrocarbons and 2-alkylcyclobutanones could be used for the detection of radiated shrimps. These studies could be applied to a variety of foods, since the concentrations of the radiation-induced hydrocarbons and 2-alkylcyclobutanones, in various foods, are different.

ACKNOWLEDGMENT

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