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2-Alkylcyclobutanones as Irradiation Dose Indicators in Irradiated Ground Beef Patties

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Alkylcyclobutanones have been recognized as chemical markers of irradiated lipid-containing foods since 1970. They are important because they are produced solely as a result of irradiation and not any other processing method. This study investigated the formation of 2-dodecylcyclobutanone (2-DCB) and 2-tetradec-5′-enylcyclobutanone (2-TDCB) in irradiated ground beef patties from commercial and noncommercial sources. Patties were irradiated using a ^{60}C source (γ-irradiation) and electron beam irradiation, at five targeted absorbed doses of 0.5, 1.0, 2.5, 5.0, and 7.0 kGy. Commercially available irradiated patties were also studied. A supercritical fluid extraction (SFE) procedure was optimized and used for the extraction and isolation of the alkylcyclobutanones. Samples can be used for extraction without a prior cleanup step, which makes this procedure rapid and convenient to use. Identification and quantitation of the cyclobutanones were done by gas chromatography−mass spectroscopy. 2-DCB was detected in all of the irradiated samples (including commercial patties), and its concentration increased linearly with the irradiation dose. Electron beam irradiation produced a greater amount of 2-DCB compared to γ-irradiation at dose levels >2.5 kGy. 2-TDCB was detected only at the two higher irradiation doses, whereas both marker compounds were not detected in the non-irradiated samples.

KEYWORDS: Alkylcyclobutanones; irradiation; gamma; electron beam; 2-dodecylcyclobutanone; 2-tetradec-5′-enylcyclobutanone; supercritical fluid extraction; gas chromatography; mass spectroscopy

INTRODUCTION

Irradiation of food is used to improve the safety and maintain the quality of foods by extending shelf life and decreasing microbial load. Since 1990 advances in the commercialization of food irradiation have led to greater international trade of irradiated foods (*1*). As a result, laws and regulations dealing with the use of food irradiation have been implemented in various countries. In the United States, the Food and Drug Administration (FDA) approved the irradiation of pork in 1985 (*2*) and that of poultry in 1990 (*3*). Approval for beef took longer and was finally given in December 1997 with maximum dose limits set at 4.5 kGy for chilled meat and at 7.0 kGy for frozen meat (*4*). Despite the technology having been around since the 1950s, consumer acceptance of irradiated food has been slow to develop (*1*). Induced radioactivity, formation of toxic byproducts, and nutrient losses in food due to irradiation are some of the concerns expressed with regard to food irradiation. Consumers, whether for or against food irradiation, demanded the right to be informed about the food they purchase. This led to irradiated food being clearly labeled as such since 1986 (*1*). In addition, there was a demand for methods capable of detecting irradiated foods and determining the amount irradiation exposure. Being able to differentiate between irradiated and non-irradiated foods is of special importance in proving the authenticity and safety of an irradiated product and for detecting mislabeled products. As the use of food irradiation as a means of preservation is increasing, a reliable and convenient method is needed to detect irradiated food and quantify the irradiation dose.

Various physical and chemical methods have been investigated for the detection of irradiated foods. Of these methods, the use of 2-alkylcyclobutanones as chemical markers of irradiation is useful for investigation of lipid-containing foods. These alkylcyclobutanones are unique radiolytic products formed from fatty acids (*5*). They are not known to be formed by cooking or any other heat-processing method, making them important as indicators of irradiation exposure.

Alkylcyclobutanones were first detected in irradiated fats by LeTellier and Nawar in 1972 (*6*). They found that when triglycerides containing C6, C8, C10, C12, C14, C16, and C18 fatty acids were subjected to irradiation, 2-substituted alkylcyclobutanones were formed as radiolytic products. These alkylcyclobutanones are cyclic compounds formed by the loss of an electron from the oxygen on the carbonyl of a fatty acid or triglyceride, followed by a rearrangement process to produce

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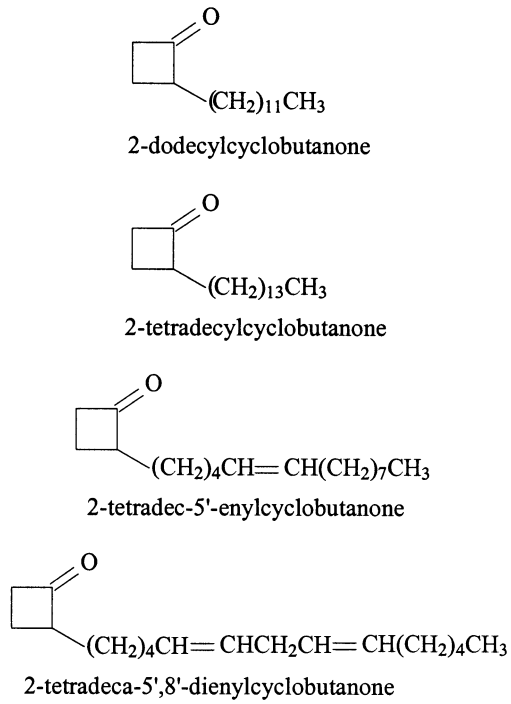

Figure 1. Basic structure and side chains of the four most common alkylcyclobutanones found in irradiated lipid-containing foods.

2-alkylcyclobutanones specific to the parent fatty acid. The resulting compounds have the same number of carbon atoms as the precursor fatty acids with an alkyl group attached at ring position 2. The four major fatty acids present in most foods are palmitic, stearic, oleic, and linoleic acids. When exposed to irradiation these acids are converted to their corresponding cyclobutanones, namely, 2-dodecyl-, 2-tetradecyl-, 2-tetradec-5′-enyl-, and 2-tetradeca-5′,8′-dienyl cyclobutanone (**Figure 1**).

Previous studies have shown that alkylcyclobutanones are formed in a variety of lipid-containing foods, including beef (*7*−*9*). Studies conducted on γ-irradiated chicken, peanuts, perilla seeds, and pork (*10*−*13*) have shown that the concentration of alkylcyclobutanones generally increases linearly with irradiation dose. However, there have been no extensive studies conducted on irradiated ground beef patties or on commercially available beef samples so far.

The most common method of isolating cyclobutanones from lipid-containing foods is solvent extraction of the lipid material followed by Florisil chromatography. This method was adopted as a European Standard (EN1785) and Ministry of Agriculture, Fisheries and Food validated method (MAFF V37) in 1996 (*14*). The method involves a long cleanup and extraction procedure and uses large volumes of organic solvents. Recent studies using supercritical fluid extraction (SFE) have shown that this procedure offsets the limitations of the Florisil method and is better suited for the extraction of the 2-alkylcyclobutanones (*8*, *15*, *16*). Thus, the main objectives of this research were (1) to optimize an SFE procedure for the isolation of alkylcyclobutanones, (2) to isolate and quantify 2-dodecylcyclobutanone (2-DCB) and 2-tetradec-5′-enylcyclobutanone (2-TDCB) in ground beef patties irradiated at different absorbed doses, and (3) to isolate and detect these two cyclobutanones in commercially available ground beef patties.

MATERIALS AND METHODS

Meat Samples. Coarse ground chubs (1.27 cm) of 80/20 trim (20% fat) were obtained from IBP, Inc. (Emporia, KS), 48−72 h post-mortem.

Table 1. Targeted and Actual Absorbed (Average) Irradiation Doses at the Gamma and Electron Beam Facilities[a]

	delivered dose (kGy)	
targeted dose (kGy)	gamma	e-beam
0.5	0.4	0.9
1.0	0.9	1.7
2.5	2.3	2.6
5.0	5.1	4.6
7.0	7.2	6.6

[a]Actual dose was measured by dosimeters at the time of irradiation as described under Materials and Methods. Values were provided by the irradiation facilities.

The trim was then ground once through a fine plate (0.32 cm) and formed into one-fourth pound patties (weight = 113 ± 3 g) using a Hollymatic patty machine (Hollymatic super model 54 food portioning machine, Hollymatic Corp., Countryside, IL). Patties were then crust frozen on aluminum trays for 15−20 min in a blast freezer (−40 °C) to facilitate handling and packaged in 3 mil nylon/polyethylene barrier pouches (Koch Supplies Inc., Kansas City, MO). Packaging oxygen diffusion rates were 3.5 g/100 in.^{2}/24 h at 70 °F (21.1 °C) and 0.6 g/100 in.^{2}/24 h at 32 °F (0 °C). Water vapor transmission rate was 0.6 cm^3/100 in.^{2}/24 h at 100 °F. The fat concentration of the patties was 18.58 ± 0.30% as measured by the microwave−solvent extraction method of AOAC International (method 985.15) (*17*). Samples were stored at −18 °C prior to irradiation. Samples were irradiated using ^{60}C (Steris-Isomedix Services, Inc., Morton Grove, IL) or electron beam (Steris-Isomedix Services, Inc., Libertyville, IL), at targeted adsorbed doses of 0.5, 1.0, 2.5, 5.0, and 7.0 kGy. Four patties were irradiated at each dose at each facility. Three patties from each batch were not irradiated and used as control patties. Two kinds of dosimeters were used for measuring the exact dose administered to the patties at the gamma facility, which included FWT-70 Opti-Chromic dosimeters (Far West Technology, Goleta, CA), for doses up to 2.5 kGy, and Harwell Red 4034 Perspex dosimeters (Oxfordshire, U.K.), for 5.0 and 7.0 kGy doses. Radiochromic dosimeters, FTW-60 series (Far West Technology), were used at the electron beam facility. The actual adsorbed doses that were achieved at the two facilities are shown in **Table 1**. The dose rates achieved were 1.01 Gy/s at the gamma facility and (2.25−3.00) × 10^3 Gy/s at the electron beam facility. Dry ice was used to ensure that samples arrived in frozen condition. Temperature through transportation was monitored using temperature data loggers (Omega Engineering, Inc., Stamford, CT). Ground beef patties obtained from a commercial source were also investigated for the formation of cyclobutanones. The patties were a one-fourth pound each (112 g) and had a fat level of 9.33% (values obtained from the Nutrition Facts label).

Fatty Acid Profile. The fat from the ground beef patties was first extracted by blending 5.00 g of the beef sample with 25 mL of hexane. The extract was passed through Whatman filter paper (no. 4) and concentrated to dryness with a rotary evaporator (temperature = 45 °C). The triglycerides were then converted to their corresponding fatty acid methyl esters (FAME) using boron trifluoride/methanol (14%), according to the procedure described by Ackman (*18*). The extracts were analyzed with a Hewlett-Packard 5890 gas chromatograph (Agilent Technologies, Palo Alto, CA) fitted with a flame ionization detector (FID) and an HP-23 cis/trans FAME column (Agilent Technologies). The GC-FID conditions were as follows: injector temperature, 250 °C; initial temperature, 60 °C; hold for 1 min; 20 °C/min to a final temperature or 195 °C; hold for 15 min; detector temperature, 260 °C; and helium carrier gas flow, 1 mL/min. The analyses were performed in triplicate.

Preparation of Patties for Extraction. All patties were stored at −80 °C prior to analysis. Once ready for analysis, the patties were tempered at room temperature for 20−30 min or until soft enough to cut. The patties were then cut into 1 cm^2 squares and immersed into liquid nitrogen. They were removed after the liquid nitrogen had stopped bubbling (~30−45 s) and ground in a Waring blender fitted with a stainless steel blending container (Fisher Scientific Co., Pittsburgh, PA).

The result was a fine homogeneous powder, which was used for the SFE procedure.

Reagents. Hexane, Florisil (60−100 mesh), anhydrous sodium sulfate, and boron trifluoride/methanol (14%) were purchased from Fisher Scientific. Wetsupport (diatomaceous earth) and sand were obtained from Isco, Inc. (Lincoln, NE). The 2-DCB standard was obtained from Acros Organics (Fisher Scientific Co.), whereas the 2-TDCB standard was custom synthesized by QuChem Ltd. (Belfast, Northern Ireland).

SFE Procedure. An Isco-Suprex prepmaster GA (Isco, Inc.) fat analyzer was used for the SFE procedure. Ground beef was homogenized with Wetsupport in a Waring blender in the ratio of 1 part beef to 2 parts Wetsupport. A 5 mL SFE cartridge (Isco, Inc.) was loaded with sand, Florisil, and ~1.5 g of the beef−Wetsupport mixture prior to being placed in the extractor. The sand protects the seals of the extraction cartridge, and the Florisil serves to trap the fat. Three beef patties were extracted for each dose level, and two cartridges were prepared per patty. The control and commercial patties were prepared and used in the same way.

Extraction was carried out under the following conditions: pressure, 340 atm; temperature, 75 °C; 5 min static and 20 min dynamic with a flow rate of CO_2 of 1 mL/min. These parameters were modified from the procedure of Tewfik et al. (*8*). The cyclobutanones were trapped on glass wool and eluted with ~25 mL of hexane. This extract was concentrated under nitrogen gas to 25 μL for control samples, commercial samples, and samples irradiated at doses 0.5−2.5 kGy and to 50 μL for samples irradiated at doses of 5.0 and 7.0 kGy. The extracts were then injected into the GC-MS.

GC-MS Analysis. GC-MS was performed with a Hewlett-Packard 5890 fitted with an HP-5 MS column (cross-linked 5% Ph Me siloxane, 30 m × 0.22 mm × 0.0.25 μm film thickness) and a Hewlett-Packard MSD 5970 detector. The flow rate for the helium carrier gas was 1 mL/min. The GC temperature program was as follows: injector temperature, 250 °C; initial temperature, 55 °C; hold for 0.5 min; ramp at 20 °C/min to a final temperature of 200 °C; hold for 1 min; ramp at 15 °C/min to a final temperature of 270 °C; hold for 1 min. The transfer line and ion source were held at 280 °C throughout the runs. The MS was set to selected ion monitoring (SIM) mode for the analysis of cyclobutanones. The ions *m*/*z* 98 and 112 were monitored for 2-DCB, and the ions *m*/*z* 98, 165, 236, and 264 were monitored for 2-TDCB. Standard solutions of 0.1, 0.25, 0.5, 0.75, and 1.0 ppm were used to calibrate standard curves for 2-DCB and 2-TDCB. The compounds were identified by comparing retention times and ion ratios with those of the standards, and the concentration in the sample was determined from the standard curve. Percent recovery for both 2-DCB and 2-TDCB was calculated by spiking control samples with 1 ppm of 2-DCB and 2-TDCB. Five grams of the control sample was spiked with 500 μL of a 10 ppm solution of the standards, run through the extraction procedure as described before, and quantified by GC-MS.

Statistical Analysis. The experimental design was a completely randomized design with a 2 × 5 factorial treatment structure. Analysis of variance and regression analysis were carried out on the data using the SAS software system release 8.1 (SAS Institute Inc., Cary, NC).

RESULTS AND DISCUSSION

Supercritical Fluid Extraction Procedure. The SFE procedure was convenient and rapid and provided a clean lipid-free extract. Because the SFE procedure functions as both an extraction and cleanup procedure (*7*), the time required for analysis is shortened considerably. Using this method, the samples can be analyzed within 3 h for irradiation treatment, compared to 2 days for the official method (European Standard, EN1785), allowing for a greater number of samples to be analyzed within a given time. The percentage recoveries were 99.48% (CV = 18.81%) for 2-DCB and 89.63% (CV = 26.71%) for 2-TDCB. Thus, these factors suggest that the SFE procedure is ideally suited for the extraction and quantitative determination of 2-alkylcyclobutanones.

Table 2. Concentration of 2-DCB in the Ground Beef Patties

	av 2-DCB concn[a] (μg/g)	
targeted dose (kGy)	gamma	e-beam
0.5	0.02 ± 0.01	0.03 ± 0.01
1.0	0.02 ± 0.00	0.03 ± 0.00
2.5	0.03 ± 0.00	0.04 ± 0.01
5.0	0.06 ± 0.04	0.12 ± 0.02
7.0	0.11 ± 0.02	0.17 ± 0.03

[a] Values are expressed in ground beef as mean ± standard deviation (n = 3).

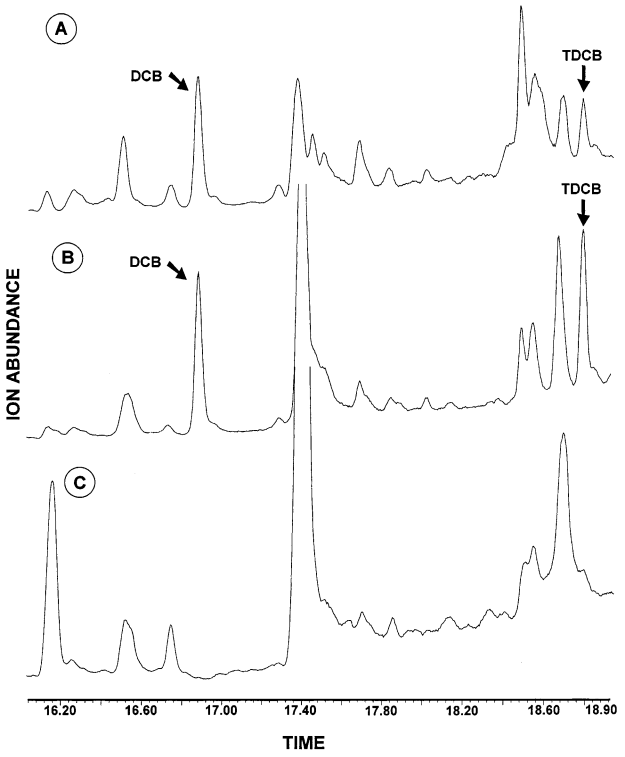

Figure 2. GC-MS chromatograms showing the detection of 2-DCB and 2-TDCB in irradiated samples: (A) 7 kGy (targeted) γ-irradiated patties; (B) 7 kGy (targeted) electron beam irradiated patties; (C) control patties. The MS was set to the SIM mode. Ions 98 and 112 were scanned for 2-DCB, and ions 98, 165, 236, and 264 were scanned for 2-TDCB.

Radiation-Induced Alkylcyclobutanones. Analysis of the fatty acid profile of the ground beef samples showed a typical composition. The samples, in grams of fatty acid per 100 g of lipid, contained 0.32 ± 0.18 C12:0, 4.59 ± 0.34 C14:0, 27.27 ± 0.35 C16:0, 5.02 ± 0.22 C16:1, 15.08 ± 0.70 C18:0, 43.44 ± 0.74 C18:1, 3.83 ± 0.67 C18:2, and 0.28 ± 0.10 C18:3. Beef fat is high in palmitic acid (27.27 g/100 g of lipid), which is a precursor to 2-DCB, and oleic acid (43.44 g/100 g of lipid), the precursor to 2-TDCB. Therefore, these two compounds were expected to be the major cyclobutanones formed in the irradiated beef patties. 2-DCB was extracted and detected in all of the irradiated samples (**Table 2**). In the commercial patties, the concentration of 2-DCB was calculated to be 0.03 ± 0.01 ppm (n = 3). 2-TDCB was detected for only the two higher doses of irradiation (5.0 and 7.0 kGy, targeted) and was not detected in the commercial samples. **Figure 2** shows the chromatograms of the 2-alkylcyclobutanones of non-irradiated and the 7 kGy (targeted) irradiated patties. The concentration of 2-DCB increased linearly ($p \geq 0.05$) with the irradiation dose for γ-irradiated and electron beam irradiated patties (**Figure 3**).

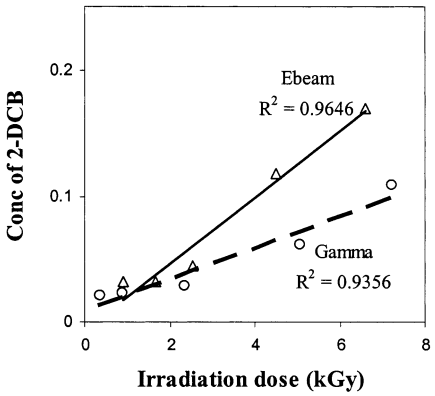

Figure 3. Response of 2-DCB (micrograms per gram of beef) with increasing irradiation dose: (△) electron beam irradiated samples; (○) γ-irradiated patties.

These results are similar to those reported for the use of 2-alkylcyclobutanones as irradiation markers in γ-irradiated peanuts, chicken, and pork (*10*−*13*), which used solvent extraction−Florisil chromatography as the purification method. Comparison of the two irradiation methods showed that the concentration of 2-DCB was higher due to electron beam radiation than γ radiation at the 2.5, 5.0, and 7.0 kGy targeted dose levels ($p \geq 0.05$). However, there was no significant difference in the concentrations formed at doses up to the targeted level of 1.0 kGy. This discrepancy in the concentrations is not easily explained. The reactions in foods irradiated with γ-rays or electron beam are mainly caused by fast-moving electrons and are essentially the same (*19*). Dose rate is thought to have an effect on the concentration of radiolytic products formed. According to previous research, a higher dose rate should typically result in a lower concentration of radiolytic products (*19*). However, as the dose rate used for electron beam irradiation was greater than that used at the gamma facility, the difference in 2-DCB levels cannot be explained on the basis of different dose rates. It is due to this difference that no attempt was made to calculate the dose administered to the commercial patties, as the irradiation history of the samples was not known. At this point the reason for the increased level of 2-DCB in the electron beam samples remains unclear.

The absence of 2-TDCB is somewhat difficult to explain as a substantial amount of precursor acid (oleic) is present in beef fat. Previous studies on radiation-induced hydrocarbons have indicated that the amount of hydrocarbons formed is inversely proportional to the chain length of the fatty acid (*20*). A similar effect could be influencing the formation of 2-TDCB. Stewart et al. (*21*) state that due to the greater fragmentation of 2-TDCB in the ion source of the mass spectrometer compared to 2-DCB, the detection limit is higher for 2-TDCB. In this study, the detection limit for 2-TDCB was 0.25 ppm as opposed to 0.05 ppm for 2-DCB. Therefore, the amount of 2-TDCB formed at the lower doses might possibly be too small to detect.

The results suggest that the SFE procedure optimized in this laboratory is a suitable alternative to the official Florisil extraction method for alkylcyclobutanones formed in ground beef. Monitoring the levels of cyclobutanones is very important from a practical standpoint, with regard to commercially available irradiated foods. Monitoring any other compounds such as hydrocarbons may not always be feasible, as they may be naturally present or formed by processes other than irradiation (*4*, *16*). Control samples, which may not be readily available in a commercial setting, would be indispensable when markers other than cyclobutanones are monitored. In contrast, controls are not required to detect cyclobutanones as it has been well established that these compounds are solely a result of irradiation processing (*5*).

This study has established that the SFE procedure can be used to extract alkylcyclobutanones in order to detect commercially available irradiated ground beef. More studies are required on commercial samples to ascertain if this method is suitable for the routine detection and quantitation of cyclobutanones in beef and other of lipid-containing foods. The increased amount of 2-DCB in the electron beam irradiated samples was an unexpected and interesting result that requires further investigation. The type of irradiation might be an important parameter in the quantification of the irradiation dose administered to commercial samples. Further research is also needed to investigate the influence of such variables as storage temperature and period and percentage of fat on the concentration of 2-alkylcyclobutanones.

ABBREVIATIONS USED

SFE, supercritical fluid extraction; 2-DCB, 2-dodecylcyclobutanone, 2-TDCB, 2-tetradec-5′-enylcyclobutanone; GC-MS, gas chromatography−mass spectroscopy; MS, mass spectrometry; MSD, mass spectral detector; PhMe, phenyl methyl.

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