Detection of cyclobutanones in non-irradiated food

Area of research interest: Chemical hazards in food and feed
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Background

Food irradiation is a processing technique that exposes food to high energy ionising radiation to improve shelf life and/or food quality. To support enforcement of legislation there is a need for reliable analytical methods capable of detecting marker compounds that are specific to the irradiation process, such as the 2-alkylcyclobutanones (2-ACBs).

Several methods to detect irradiation are available and for fatty foods the standardised European Committee for Standardization (CEN) method EN1785 is based on measurement of 2-ACBs that are produced by the irradiation of lipids. There have been advances in analytical techniques and new methods of isolating and detecting 2-ACBs are available.

Improvements to method EN1785 are required to lower detection limits of 2-ACBs. This will aid in an investigation into whether 2-ACBs can be detected in non-irradiated food or in food processed by means other than irradiation.

Improvements could also widen the scope of the method and permit detection of the use of irradiated ingredients in composite foods, where lower detection limits will be required.

Conversely, lowering of the limit of detection would increase the need to confirm the absence of 2-ACBs in foods that have not been irradiated and possibly derive a threshold value to distinguish irradiated from non-irradiated foods.

Research Approach

Literature review: Literature review describing the chemistry of 2-ACB formation in irradiated foods along with an overview of the analytical methods currently available for the determination of 2-ACBs. The performance characteristics, including cost and practical use, of the available methods were compared.

Compare analytical approaches for the detection of 2-ACBs: The detection and quantification limits of each of the analytical approaches investigated (GC-MS, LC-MS/MS with and without derivatisation) were compared. This considered the cost and practical application of the methods. The most sensitive method was identified.

Model studies: Studies were carried out by treating samples of palmitic acid, glyceryl tripalmitate and 1,3-dipalmitoyl-2-oleoylglycerol with non-irradiation based food processing techniques. They

were heated for various times at different temperatures in the presence and absence of oxygen, light, and redox active metal salts. They were also heated in a microwave oven, frozen and then defrosted in the microwave oven and heated to different temperatures in a pressure cooker. The processed fats were extracted, derivatised and analysed by LC-MS/MS.

Results

The literature review concluded that a significant amount of work has been reported describing the detection of 2-ACBs in a variety of irradiated foods and a number of inter-laboratory trials have been carried out successfully. There is much scope for improving sensitivity of the methods so that lower levels of 2-ACBS can be detected.

Knowledge is lacking regarding the true performance of the methods currently used, the optimum procedures for separating 2-ACBs from fat, the limits of detection of irradiated ingredients in foods, the relative formation rates of 2-ACBs from fatty acids in different parts of triacylglycerols, the longevity of 2-ACBs on storage, their fate on food processing, and the possible presence of 2-ACBs in non-irradiated foods.

Methods of analysis for 2-DCB, the only commercially available 2-ACB, have been developed by direct measurement of 2-DCB, and following derivatisation, by GC-MS and LC-MS techniques. Both GC-MS (without derivatisation) and LC-MS/MS (with derivatisation) would be available in most food testing laboratories, both have a similar limit of detection (approximately 1 ng/mL) and both are suitable for the low level detection of 2-DCB.

Model studies looked at the effects of dose by different irradiation sources on fatty acids and triacylglycerols. The only 2-ACB detected was 2-DCB. This may have been because of a low irradiation yield or the instrument sensitivity not being high enough to enable detection. This highlights the need for authentic standards to allow method optimisation, as 2-DCB was the only 2-ACB currently available and used for method development.

Studies were also carried out on a number of non-irradiation based processing techniques. No peaks attributed to 2-DCB were detected in any of the samples. This is not a surprising result as there have been no previous reports of 2-ACBs formed from non-irradiation based processing techniques, to the authors' knowledge.

Published Papers

1. Crews, C., Driffield, M. & Thomas, C. (2012) Analysis of 2-alkylcyclobutanones for detection of food irradiation: current status, needs and prospects doi: doi.org/10.1016/j.jfca.2011.11.006

Research report

England, Northern Ireland and Wales

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