

## Thermoluminescence analysis can identify irradiated ingredient in soy sauce before and after pasteurization



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### ABSTRACT

Thermoluminescence (TL) analysis was conducted to identify small quantities (0.5%, 1%, and 1.5%) of  $\gamma$  ray- or electron beam-irradiated garlic powder in a soy sauce after commercial pasteurization. The sauce samples with  $\gamma$  ray- and electron beam-irradiated (0, 1 or 10 kGy) garlic powder showed detectable TL glow curves, characterized by radiation-induced maximum in the temperature range of 180–225 °C. The successful identification of soy sauces with an irradiation history was dependent on both the mixing ratio of the irradiated ingredient and the irradiation dose. Post-irradiation pasteurization (85 °C, 30 min) caused no considerable changes in TL glow shape or intensity. Interlaboratory tests demonstrated that the shape and intensity of the first TL glow curve (TL1) could be a better detection marker than a TL ratio (TL1/TL2).

### 1. Introduction

Soy sauce is a liquid made from soybeans or a combination of soybeans and wheat. It is rich in nutrients such as amino acids, oligosaccharides, and vitamins. Due to the widespread popularity of soy sauce in many countries including Japan, Korea, China, and others, various types of blended or mixed soy sauces that contain spices, herbs, and fish ingredients with bioactive compounds are commercially available (Kim et al., 2015). Ingredients like garlic, red chili, black pepper, turmeric, seafood, and meat are often added to various commercial soy sauces to improve sensory qualities and consumer acceptance (Kim and Lee, 2008). However, dried spices are prone to microbial contamination during sun drying, storage, and transportation (Farkas, 1988; Lilie et al., 2007).

Irradiation is one of the currently employed methods for microbial decontamination of spices without adversely affecting flavor quality (Farkas, 1988). To achieve microbiological safety, these spices are exposed to an average radiation dose of 10 kGy (Variyar et al., 1998) before being mixed into food products. The safety, wholesomeness, and nutritional adequacy of irradiated foods are now well documented and accepted by all major health and food authorities (WHO, 1999). To improve food hygiene and quality, irradiation technology is approved

in over 50 countries and is commercially practiced in more than 30 countries around the world (IAEA, 2013). However, various national and international regulations with mandatory labeling requirements restrict the general use of this technology. In the commercial domain of international trade, the acceptability of irradiated foods demands reliable detection methods to enforce regulations and traceability (Chauhan et al., 2009). Therefore, it is essential to develop reliable methods for distinguishing between irradiated and non-irradiated foods.

Several detection methods have been developed for the identification of irradiated foods (Delincée, 1998; Codex, 2003; Sanyal et al., 2009; Ahn et al., 2012a; Kwon et al., 2013). The European Union has accepted ten different methods (Chauhan et al., 2009), all of which are endorsed by the Codex commission (Codex, 2003). TL measurement is a promising technique because of its sensitivity and efficiency in detecting irradiation history even after prolonged storage (EN1788, 2001). This method can be employed for food materials from which silicate minerals can be isolated (Sanderson et al., 1996). The success of this technique depends on the quantity and quality of the isolated polyminerals, which contain various constituents in different amounts, namely quartz, feldspar, and clay particles (Sanderson et al., 1996; Correcher et al., 1998; EN1788, 2001; Sanyal et al., 2009; Kwon et al.,

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2013). TL outputs from irradiated samples exhibit variable intensities and diverse TL glow curve structures. According to the EN 1788 (EN 1788, 2001), detection efficiency can be improved by exposing the samples to a normalization dose of 1 kGy, followed by the measurement of a second TL glow. However, the presence of extremely low quantities of contaminants or irradiated ingredients in foods makes the detection process challenging (Delincée, 1998). In addition, only limited information is available on the efficiency of TL detection after electron beam irradiation and the effect of various post-irradiation processing techniques.

The main objective of this study was to identify the commercially available mixed soy sauces containing low quantities of  $\gamma$  ray-or electron beam-irradiated garlic powder using TL analysis. The effect of soy sauce pasteurization on TL detection markers was also investigated. Independent interlaboratory tests were performed to assess the efficacy of this technique for use in routine analysis of mixed sauces.

## 2. Materials and methods

### 2.1. Sample preparation and irradiation

Garlic powder (garlic 100%, >80 mesh, YS Co., Korea) was purchased from a local mart and has a moisture content closer to 11.0%. The sample was irradiated at absorbed doses of 0, 1, and 10 kGy (dose rate: 2.1 kGy/h) for disinfection and microbial decontamination using a Co-60  $\gamma$ -ray source (AECL, IR-79, MDS Nordion International Co. Ltd., Ottawa, Ontario, Canada) at the Korean Atomic Energy Research Institute (KAERI) in Jeongseup, Korea. Electron beam irradiation was carried out using an electron accelerator (High Energy Linear Accelerator, 10 MeV, EB Tech, Daejeon, Korea). The absorbed doses were assured using alanine dosimeters with a diameter of 5 mm (BRUKER, Rheinstetten, Germany) (uncertainty:  $\pm 5.4\%$ ), which were attached to the sample cart, while EPR signal intensities were measured by the Bruker EMS 104 EPR analyzer (BRUKER,

Rheinstetten, Germany).

### 2.2. Sauce preparation

Commercial soy sauce products (C Co., Korea) were obtained from local mart and the samples were proved to be not irradiated through pre-experiment (TL). Mixed soy sauce samples were prepared by combining Korean fermented soy sauce with non-irradiated (0 kGy) or irradiated (1 or 10 kGy) garlic powder at blending ratios of 0%, 0.5%, 1.0%, and 1.5% at C Foods Company (Seoul, Korea), based on commercial practices. The prepared samples were used for TL detection studies before and after pasteurization (85 °C, 30 min) to determine the effect of heat processing on the detection sensitivity of radiation-induced TL glow signals.

### 2.3. TL measurements

Two detection laboratories equipped with different thermoluminescence dosimetry (TLD) readers for irradiated foods participated in the interlaboratory validation test: Kyungpook National University (KNU) using a TLD-4500 Harshaw instrument (Harshaw, Erlangen, Germany) and KAERI using a DK/TL/OSL-DA-20 RISØ instrument (National Lab, Roskilde, Denmark).

The EN 1788 method (EN 1788, 2001) was employed to separate the minerals from the samples (100 g) by density separation. The separated minerals were mounted on clean stainless steel discs and kept overnight in a drying oven at 50 °C to remove low-temperature peaks. The discs containing minerals of control and irradiated samples were weighed to determine the quantity of minerals deposited. Nitrogen was flushed in the heating chamber to reduce spurious TL arising due to the presence of oxygen. TL measurements were conducted using a TL reader at 50–400 °C with a heating rate of 6 °C/s. According to the EN 1788 (EN 1788, 2001), detection efficiency can be improved by exposing the samples to a normalization dose of 1 kGy, followed by the measurement of a second TL glow. Thus, after measurement of glow 1 (TL1), the discs with the

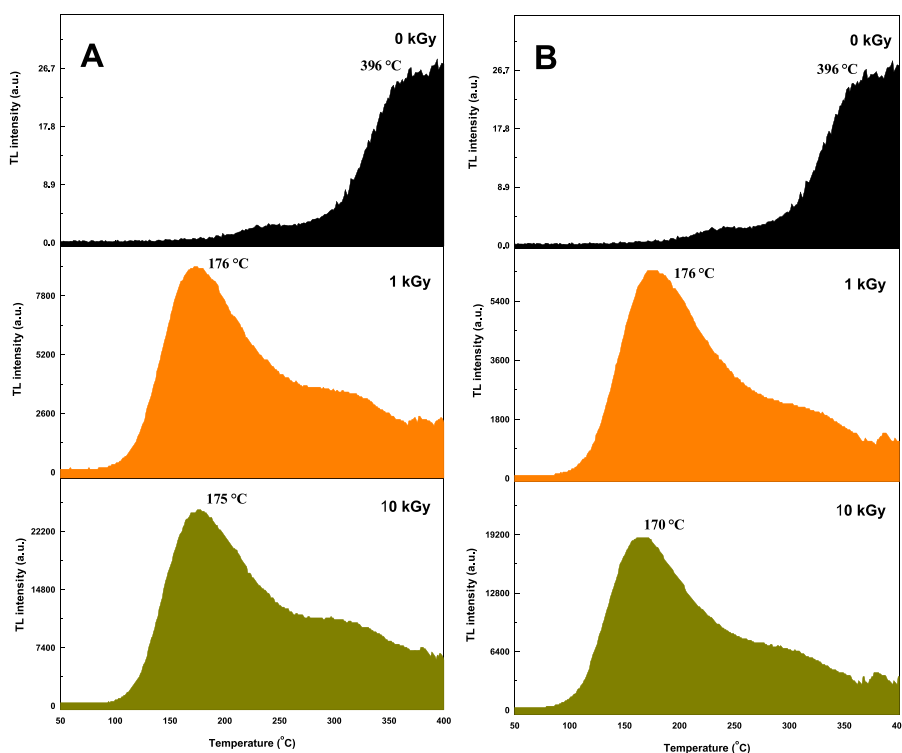


Fig. 1. TL glow curve of minerals separated from  $\gamma$  (A)- and electron beam (B)-irradiated garlic powder.

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