

## **Properties of Thermoluminescence according to Importation Area of Sesame and Perilla Seeds Treated with Irradiation**

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### **Introduction**

As commercialization of irradiation technology increased, demand for a convenient and reliable method for detection of irradiated foods is growing. A number of methods have been investigated over years, and detection methods using electron spin resonance (ESR) or thermoluminescence (TL) have shown significant potential. TL technique, where heat is used to stimulate the release of trapped energy in the form of light, is relatively simple. The dried material is heated at a constant rate of 5~10°C /s, up to a final temperature of 300~400°C, and the light emission is registered by a sensitive detector. The TL technique has shown potential for the identification of various irradiated foods. In Korea, sesame and perilla seeds are used as an edible oil and seasoning and are sometimes imported from China or Sudan for the control of demand and supply. These seeds are particularly susceptible to be contaminated microorganisms, which major result in a decrease in quality control and shelf-life, and a public health hazard because of improper handling and poor quality control. Therefore, currently, although irradiation for sesame and perilla seeds is not permitted within and out a country, the use of ionizing radiation for imported sesame and perilla seeds could be considered as an effective treatment against possible microbial contamination and thereby increase safety assurance. With necessity of irradiation, detection technique for these also must established to provide an opportunity of choice to the consumers and control international trade.

Based on this background, the main object of this article was to establish an optimized TL method through intensity, maximum temperature, and shape and ratio of first and second glow curve in TL and observe properties of TL according to importation area of irradiated sesame and perilla seeds.

### **Materials and Methods**

#### **Materials and irradiation**

Chinese sesame and perilla seeds and Sudanese sesame seed were purchased from a local market in Taejon, Korea. Samples (250 g) were paked in polyethylene bags and irradiated by a Co-60 irradiator (AECL, IR-79, Canada) at 1, 5, and 10 kGy with a dose rate of 166.6 Gy/min. Total

absorbed dose was determined using a ceric-cerous dosimeter.

#### **Measurement of Thermoluminescence (TL)**

Separation of mineral was carried out through methods of DIN EN 1788. The dried minerals (1mg) were deposited onto a clean stainless steel disc (10 mm diameter, 0.5 mm thickness) and fixed with silicon solution, which mixed silicon rubber (LDC 210, Dow Corning Korea Ltd, Seoul, Korea) and hexen in the ratio of 5 : 1, respectively. Then, samples fixed on the stainless steel disc with the silicon solution dried and measured with a thermoluminescence (TL) reader. TL measurement was carried out using a TL reader (Harshaw 3500, Wermelskirchen, Germany) under heat ranging from 50 to 320°C at a rate of 6°C /s and held at 320°C for 10 s. The light emission was recorded in a temperature-dependent mode as a glow curve in units of nano coulombs (nC). After the first glow curve was measured, the discs with the minerals were subsequently re-irradiated under a Co-60 irradiator at a normalizing dose of 1, 5, and 10 kGy. The TL intensity was re-measured again after the re-irradiation step (second glow curve). The glow curve ratios G I (G1) first glow curve of unirradiated sample/second glow curve of irradiated sample at 1, 5, and 10 kGy) and G II (G2, G3, G4) [first glow curve per irradiation dose (G2=1 kGy, G3=5 kGy, G4=10 kGy)/second glow curve of irradiated samples at 1, 5, and 10 kGy] were then determined. TL measurements of all samples were repeated three times.

#### **Evaluation by the shape of glow curve and glow curve ratio**

According to DIN EN 1788, glow curve ratios of irradiated samples are typically greater than 0.5, whereas those of unirradiated samples are generally below 0.1. If glow curve ratios between 0.1 and 0.5 are obtained, interpretation of the glow curve shapes is needed to decide whether the sample has been irradiated or not, since the shapes of the first glow curve appear in higher temperature region than those of the second glow curve. Therefore, the definition above was applied in this research.

#### **Statistical analysis**

Significant differences were determined using Duncan's multiple range test in a one-way ANOVA with SPSS (Statistical Package for Social Science) version 7.5. All experiments were repeated three times.

#### **Results and Discussion**

The TL intensities measured immediately after the irradiation for the minerals from Chinese sesame and perilla seeds and Sudanese sesame seed were  $46.7 \pm 3.9$ ,  $74.7 \pm 6.6$ , and  $30.1 \pm 7.1$ , for the

unirradiated samples and  $8,568.7 \pm 870.2$ ,  $6,207.3 \pm 1,439.3$ , and  $1,625.5 \pm 278.2$  nC for 10 kGy irradiated samples, respectively. TL intensities were in the order of Chinese perilla seed > Chinese sesame seed > Sudanese sesame seed at 1 and 5 kGy but, at 10 kGy, Chinese sesame seed > Chinese perilla seed > Sudanese sesame seed. Irradiated Chinese sesame and perilla seeds exhibited higher TL intensities than the irradiated Sudanese sesame seed. This difference in TL observed between both samples seemed to be caused by the differences in mineral composition, size, and amount used for the measurement. However, since irradiated samples showed higher TL intensities than the unirradiated samples in all the samples, detection of irradiation was possible by noting the difference in TL intensities. Correlation equations and coefficients for irradiated Chinese sesame and perilla seeds and Sudanese sesame seed showed straight-line relationships and very high correlation coefficients between irradiation dose and the corresponding TL intensity. Significant differences were shown between irradiation dose and TL intensity ( $p < 0.05$ ). TL intensities of the second glow curves in Chinese and Sudanese sesame seeds increased with increasing re-irradiation dose but in Chinese perilla seed, there was no difference of TL intensities between 5 and 10 kGy. Since the glow curve ratios G I (G1) were below 0.05, they were classified as unirradiated samples on definition of DIN EN 1788. Although glow curve ratios G II (G2, G3, G4), which investigated on increasing re-irradiation doses of irradiated samples exhibited a decrease with increasing re-irradiation dose, since these were above 0.5 in several glow curve ratios, detection was possible though those on definition of DIN EN 1788. Maximum TL temperatures of the first glow curves for the minerals separated from irradiated Chinese sesame and perilla seeds and Sudanese sesame seed were between  $171.6 \pm 4.3 \sim 178.3 \pm 7.5^\circ\text{C}$ ,  $179.7 \pm 2.1 \sim 181.0 \pm 2.1^\circ\text{C}$ , and  $166.9 \pm 0.7 \sim 171.9 \pm 2.1^\circ\text{C}$ , and those of the second glow curves were between  $147.9 \pm 1.4 \sim 153.8 \pm 5.2^\circ\text{C}$ ,  $142.9 \pm 0.8 \sim 147.2 \pm 2.6^\circ\text{C}$ , and  $146.6 \pm 2.7 \sim 147.5 \pm 3.4^\circ\text{C}$ , respectively. Therefore, detection was possible through the comparison of maximum TL temperatures since those of second glow curves appeared at a lower temperature range than those of the first glow curves. Maximum TL temperatures of all irradiated samples were below  $190^\circ\text{C}$ , which are within the temperature ranges of  $150 \sim 250^\circ\text{C}$  recommended by DIN EN 1788. All irradiated samples normally showed a unique first glow curve between 150 and  $250^\circ\text{C}$ , but was not found in the unirradiated samples. In addition, since the shape of the second glow curves showed at a lower region than those of the first glow curves, detection of irradiated samples was possible through the position of glow curve shapes. Moreover, as the irradiated samples showed glow curve ratios below 0.5, they were classified as irradiated samples. Therefore, these results based on the data above suggest that detection of irradiated Chinese sesame and perilla seeds and Sudanese sesame seeds is possible through the examination of the TL intensity, glow curve ratio, maximum TL temperature, and the shape of glow curves of TL measured for miners separated from the unirradiated and irradiated samples.

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