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# DEVELOPMENT OF PHOTOSTIMULATED LUMINESCENCE TECHNIQUE FOR DETECTING IRRADIATED FOOD

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## *ABSTRACT*

*The exposure of food to ionizing radiation is being progressively used in many countries to inactivate food pathogens, to eradicate pests and to extend shelf-life of food. To ensure free consumer choice, irradiated food will be labeled. The availability of a reliable method to detect irradiated food is important to enforce legal controls on labeling requirements, ensure proper distribution and increase consumer confidence. This paper reports on the preliminary application of photostimulated luminescence technique (PSL) as a potential method to detect irradiated food and perhaps be used for monitoring irradiated food on sale locally in the near future. Thus this study will be beneficial and relevant for application of food irradiation towards improving food safety and security in Malaysia.*

**Keywords**: Detect irradiated food, food irradiation, labelling requirements, legal controls, luminescence technique,

## INTRODUCTION

Treatment of food with ionizing radiation can contribute to food safety (i.e. to prevent avoidable food-borne diseases) and to food security (i.e. to make more food available to more people, eventually at lower cost). Safety of the treatment has been recognised by international organizations such as FAO, WHO and Codex Alimentarius Commission. To date, health and safety authorities in over 60 countries worldwide have approved the irradiation of over 60 kinds of foodstuffs. Global production of irradiated foods, while still small in volume, has increased steadily in recent years, from 300,000 tonnes in 2000 reaching to 405,000 tonnes in 2005 (Kume et al., 2009). The main countries applying this technology include Belgium, Brazil, China, France, South Africa and USA with main applications being disinfection of spices and dry vegetables, disinfestation of grains and fruits, disinfection of meat and seafood, and sprout inhibition.

In view of the growing interest in food irradiation, the development of analytical methods for correct identification of irradiated samples from non-irradiated samples has thus become important for upholding regulatory controls, checking compliance against labeling requirements, facilitating international trade, and reinforcing consumer confidence. Regulatory authorities in all countries are interested in having reliable methods to detect irradiated foods.

Since 1980, extensive research was undertaken in many countries especially in Europe which resulted in the development of a range of test methods that can be used to reliably determine the irradiation status of a wide variety of foods (Delincee, 2002). A milestone was reached in 1996, when five European Standards were adopted by the European Committee for Standardization (CEN) as shown Table 1. In 2004, 5 more validated standard methods came into existence. The methods used for the detection of irradiated foods are based on physical, chemical, biological, and microbiological changes in food products during irradiation, although these changes are minimal.

Table 1: European standards for detection of irradiated food

| **Standards no.** | **Method** | **Validated products** |
|---|---|---|
| EN 1784 | Gas chromatographic (GC) analysis of hydrocarbons | Chicken, pork, beef, avocadoes, mangoes, papayas, camembert |
| EN 1785 | GC/MS analysis of 2-alkylcyclobutanones | Chicken, pork, liquid whole egg |
| EN 1786 | ESR spectroscopy of bones | Chicken, fish, frog legs |
| EN 1787 | ESR spectroscopy of cellulose | Paprika powder, pistachio nut shells, strawberries |
| EN 1788 | Thermoluminescence of silicate minerals | Herbs and spices, shrimps (to be added in revised version: Shellfish in general, fresh or dehydrated fruits and vegetables, potatoes) |
| EN 13708 | ESR spectroscopy of crystalline sugars | Dried papayas, dried mangoes, dried figs, raisins, herbs and spices, shellfish |
| EN 13751 | Photostimulated luminescence | Herbs and spices, shellfish |
| EN 13783 | Microbiological screening using direct epifluorescent filter technique/aerobic plate count (DEFT/APC) | Herbs and spices |
| EN 13784 | DNA comet assay screening | Chicken, pork, plant cells, e.g. seeds |

Source: Delincee (2002)

The luminescence method for detecting irradiated food is one of the standard methods approved by Codex Alimentarius Commission and adopted as European Standard EN 1788 and EN 13751. The term luminescence applies to the emission of light from irradiated solids based on the effect that a small portion of absorbed radiation energy stored at low temperature is emitted in the form of light. The intensity of the emitted light can be measured and detected by either a photostimulated luminescence (PSL) or thermoluminescence (TL) detector as photon counts. This method is found suitable for food products such as herbs, spices, bulbs, tubers, vegetables, cereals, shellfish, and fruits containing silicate minerals. A PSL detector is shown as in Figure 1.

Figure 1: Photostimulated luminescence (PSL) detector

The initial work using luminescence was reported for whole samples of spices (Sanderson et al., 1989). The signal intensity is dependent on applied irradiation dose and temperature during irradiation. Correcher et al. (1998) reported that this method could also be used to differential between irradiated and non-irradiated paprika. The signal is long-lived and remains reliably greater in irradiated samples than in control samples over many months but diminished with time after irradiation (Autio and Pinnioja, 1990). Other researchers have found that luminescence technique is a promising method for identifying chicken, fish, herbs, red pepper, garlic, ginger powder, powdered dry leaf vegetable, dried mushroom, dried fruits treated with irradiation (Atta et al., 2001; Goto et al., 2005; Khan et al., 2002; Kim et al., 2012; Malec-Czechowska et al., 2003; Sukdeb et al., 2010). This method can be applied for the detection of irradiation process for a large variety of food which silicate minerals can be isolated. Its detection limit and sensitivity depend on the quantity of minerals and the way of recovery from the irradiated sample as well as suitability of optimum threshold selected for the analysis.

In Malaysia, limited research was conducted on identifying irradiated potatoes, and fruits by impedance spectroscopy and detection of irradiated spices using viscosity but varying responses were observed with different food samples (Zainon and Muhamad Lebai Juri, 2000). In the present study, we will report on PSL screening of irradiated and non-irradiated food samples. We will demonstrate the signals can be used as an early indicator that reflects the irradiation history of the food samples.

## MATERIALS AND METHODS

### Preparation of Samples

Samples in powder form (garlic powder and seasonings) were dispensed into 50 mm Petri dishes to cover the base in a thick layer; prawn samples were dispensed as intestines; rice, white and black pepper samples were dispensed as whole grains or granules; garlic, potato; onion were dispensed as slices of their outer skin or roots (parts contain the most silicate minerals) and dry noodle was crushed and dispensed in small pieces. Samples were dispensed in subdued lighting to minimize bleaching and under a laminar flow cabinet to minimize cross-contamination. Initial or screening measurements were made on duplicates, followed by irradiation with 1 kGy, then a second measurement (CalPSL) after overnight storage in the dark.

The samples were subjected to gamma irradiation at either commercial cobalt-60 irradiator Sinagama in Malaysian Nuclear Agency or Gamma-cell 220 in Universiti Kebangsaan Malaysia (UKM). Dosimetry was performed using aqueous Fricke (ASTM Standard: E 1026, 2004) and ceric-cerous dosimeter.

**PSL Measurement**

Measurements were conducted for 60 s with signals recorded every second and as a cumulative terminal count. Lower threshold of 700 (T1) and upper threshold of 5000 (T2) counts for 60 s were set to separate negative, intermediate, and positive categories. Results were to be recorded as PSL (recording each separate count) and summary (terminal counts only) files for both screening and calibrated measurements, leading to classification as non-irradiated, irradiated, or requiring further investigation.

Classification of samples using screening mode was based on a consensus of the duplicates. A sample that gives two positive (>T2) results should therefore be classified as irradiated. Samples which gave intermediate signals for both duplicates should be classified as intermediate and requiring further investigation. A sample that gives two negative (<T1) results should therefore be classified as non-irradiated.

## RESULTS AND DISCUSSIONS

Figure 2 showed PSL signals before and after calibrating dose of 1 kGy. In screening mode (initial PSL counts), good separation was observed between the irradiated and non-irradiated samples. As previously mentioned in report on PSL measurements for different foods, photon counts of less than 700 were normally detected in the non-irradiated foods, while irradiated foods showed higher than 5000 photon counts (European Standard, 2002; Sanderson et al., 1998). In this study, when the threshold is applied, all six non-irradiated material showed signals below the lower threshold whereas five of six irradiated material showed signals greater than the upper threshold; however one irradiated sample (garlic powder) produced signals between the lower, T1 and upper threshold, T2 (intermediate). This finding suggests there may be some scope for product-specific threshold due to the geographical origin of the garlic powder (Sanderson et al., 2003).

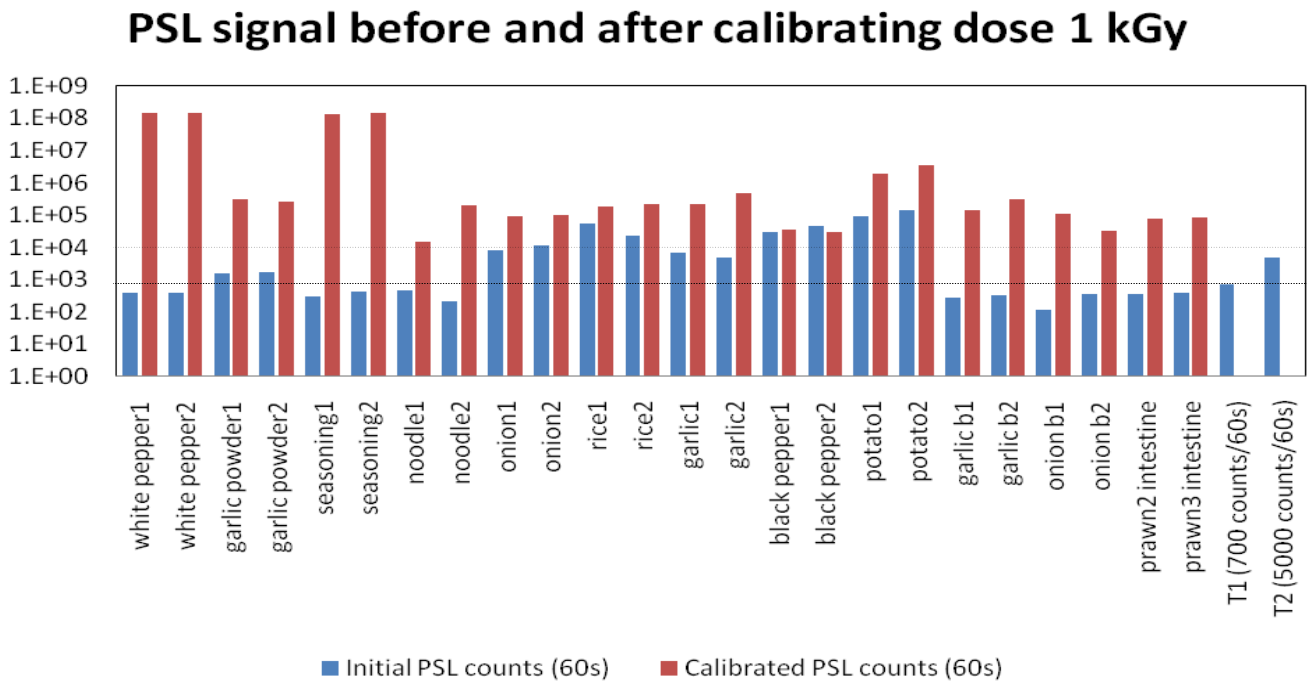


Figure 2: Initial PSL signal and calibrated signal from duplicated samples. Last two lanes are threshold signals, T1 and T2 used as reference for classification decision

Calibration of all samples (re-irradiation at 1 kGy) produced calibrated PSL counts greater than the upper threshold, when plotted against screening results for the same samples clearly confirm the status of the samples as separated by partitions shown in Figure 3. Table 2 listed those samples as white pepper, seasoning, noodle, garlic b, onion b, and prawn intestine were separated into neg-pos (negative-positive) group and classified as non-irradiated samples. Pos-pos (positive-positive) group however contained samples considered as irradiated and they include onion, rice, garlic, black pepper and potato. Garlic powder was the only sample in the int-pos (intermediate-positive) group and classified as a sample likely to be irradiated or contained component that has been irradiated and therefore require further investigation by TL analysis or other validated methods.

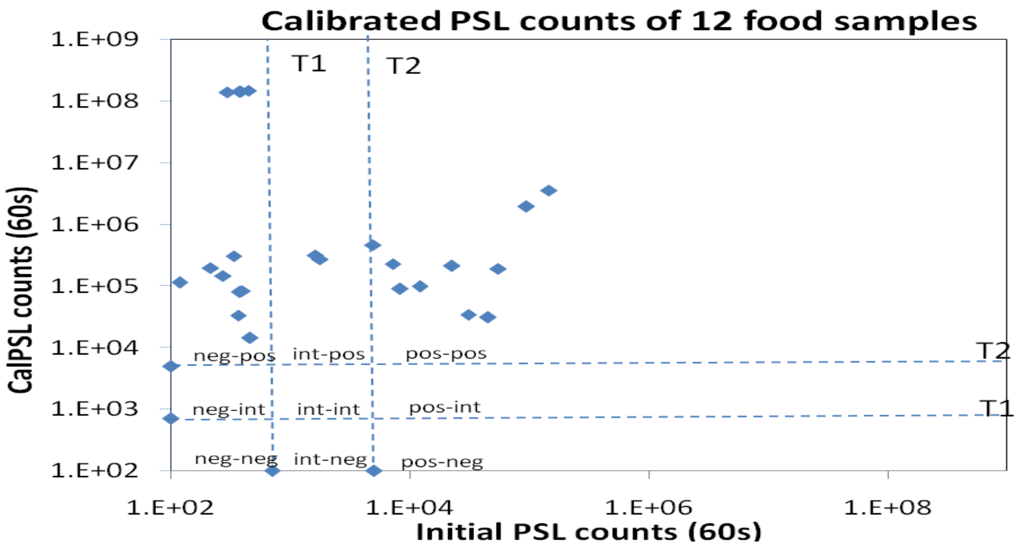


Figure 3: Calibrated PSL results versus screening results for all the samples tested. T1 and T2 are lower and upper threshold, respectively

Table 2: Qualitative results for all samples*

| **Samples** | **Initial screening** | | | **Classification of sample** |
|---|---|---|---|---|
| | **Negative** | **Intermediate** | **Positive** | |
| White pepper | 2 | 0 | 0 | Non-irradiated |
| Garlic powder | 0 | 2 | 0 | Likely to be irradiated or contains irradiated component (Need further investigation with TL or other validated methods) |
| Seasoning | 2 | 0 | 0 | Non-irradiated |
| Noodle | 2 | 0 | 0 | Non-irradiated |
| Onion | 0 | 0 | 2 | Irradiated |
| Rice | 0 | 0 | 2 | Irradiated |
| Garlic | 0 | 0 | 2 | Irradiated |
| Black pepper | 0 | 0 | 2 | Irradiated |
| Potato | 0 | 0 | 2 | Irradiated |
| Garlic b | 2 | 0 | 0 | Non-irradiated |
| Onion b | 2 | 0 | 0 | Non-irradiated |
| Prawn intestine | 2 | 0 | 0 | Non-irradiated |

*All samples showed positive result after re-irradiation of 1 kGy (Calibrated PSL)

## CONCLUSIONS

PSL detection methods can be used to detect the irradiation treatment of the analyzed food samples. It is rapid, does not require separating the minerals from the foods and also convenient as a preliminary screening method to detect irradiated foods. The availability of the method to detect irradiated food in Malaysia will be useful to the Ministry of Health for enforcing labeling control in Malaysia. The use of calibration must be recommended where CalPSL can either confirm or increase the percentage of correct classification. Further investigation is recommended for all cases where calibration does not resolve the issue.

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