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The Use of Pulsed Photostimulated Luminescence (PPSL) and Thermoluminescence (TL) for the Detection of Irradiated Perilla and Sesame Seeds

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Abstract

To establish a detection method of irradiated perilla and sesame seeds, studies were performed with pulsed photostimulated luminescence (PPSL) and thermoluminescence (TL). The PPSL photon counts of the mineral separated from irradiated sesame and perilla seeds were higher than unirradiated ones and exhibited an increase with increasing irradiation dose and mineral content. Also TL intensities of minerals separated from irradiated sesame and perilla seeds increased with increasing irradiation dose. In all samples, detection was possible with shapes and maximum TL temperatures of the second glow curves showing lower regions than those of the first glow curves and correctly classified as irradiated samples. Glow curve ratios of irradiated samples were higher than 0.5. These results suggest that PPSL and TL are applicable methods for the detection of irradiated perilla and sesame seeds.

Key words: pulsed photostimulated luminescence (PPSL), thermoluminescence (TL), perilla and sesame seeds, gamma irradiation

INTRODUCTION

Pulsed photostimulated luminescence (PPSL) and thermoluminescence (TL) are radiation-specific phenomena from energy stored by trapped charge carriers following irradiation (1). Releasing such stored energy by thermal or optical stimulation can result in a detectable luminescence emission (2-5). Most foods contain mineral debris, typically silicates, or bioinorganic materials. When exposed to ionizing radiation, these materials store energy by trapping charge carriers at structural, interstitial or impurity sites. Subsequent stimulation by electromagnetic radiation of the appropriate wavelength releases the trapped charge carriers, resulting in the emission of pulsed photostimulated luminescence (PPSL) with characteristic emission spectra (6). PPSL has been mainly developed at the Scottish Universities Research and Reactor Center (SURRC) as a detection method which is designed to allow direct measurements for rapid screening purposes without the need for sample preparation or re-irradiation such as the TL of irradiated foods (7-9). It is also able to examine inorganic systems in the presence of organic matter. The PPSL detection method has been studied as a rapid screening method for many irradiated foods including the bown shrimp (1), white ginseng powder (6), pepper powder, dried herbs, fresh shrimp, potato, soybean, dried fig, chestnut, dried squid, and dried cod (7). Thermoluminescence (TL) has been used as an irradiation-detection technique (10-17) for various foods such as spices, herbs, fruits, vegetables, and seafoods and, specifically, shown as an applicable method for the detection of all irradiated spices (18-26). Based on this previous research, this study was carried out to establish PPSL and TL methods for detecting irradiated perilla and sesame seeds.

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MATERIALS AND METHODS

Materials and irradiation

Perilla and sesame seeds harvested in Korea were purchased from a local market. Samples were packed in polyethylene bags and were irradiated using a Co-60 irradiator (AECL, IR-79, Ontario, Canada) with 1, 5, and 10 kGy, with a dose rate of 10 kGy/h at the Korea Atomic Energy Research Institute. To measure the exact total absorbed dose of gamma irradiation, the dose rates for cobalt-60 sources were determined using a ceric-cerous dosimeter.

Preparation of mineral extract

The 100 g samples were agitated with 2 L water for 5 min. The suspended samples were filtered through a 250-μm nylon cloth, and the constituents retained were discarded. The filtered solution was allowed to settle for about 5 min to separate the sediment minerals from the supernatant. The sediment minerals were suspended in 5 ml sodium polytungstate [$Na_6O_{39}W_{12}\,H_2O$] (Fluka 71913) solution which had been adjusted to a density of 2.0 g/ml by an addition of water for the separation of minerals and adhering organic materials. The minerals were pelleted through centrifugation for 2 min at 1000 rpm after a 5-min ultrasonic treatment. The low-density layer was decanted. This procedure was repeated until all the organic materials were removed. After the polytungstate solution was removed, the minerals were washed twice in water and pelleted through centrifugation at 1000 rpm, followed by a 10-min treatment with 1 M HCl to remove carbonates. After neutralizing with 1 M NH_4OH for 10 min, the solution was discarded. The minerals were washed twice in deionized water and centrifuged at 1000 rpm for 2 min to separate a mineral fraction. After the supernatant was decanted, the remaining water was then rinsed off with 3 ml of acetone twice and dried in a laboratory oven at 50°C for 3 h. The dried minerals (1 mg) were deposited onto a clean stainless steel disc (10 mm diameter, 0.5 mm thickness), fixed with silicon spray, then dried and measured with a thermoluminescence (TL) reader (27).

Measurement of pulsed photostimulated luminescence (PPSL)

The PPSL system purchased from SURRC is composed of a control unit, sample chamber, and detector head assembly, and the control unit contains a stimulation source which is comprised of an array of infrared light (880~940 nm) emitting diodes which are pulsed symmetrically on and off for equal periods. PPSL is detected by a bialkali cathode photomultiplier tube operating in the photon counting mode. Optical filtering is used to define both the stimulation and detection wavebands. The separated minerals (1, 5, 10 mg) were introduced in 50 mm diameter disposable petri dishes (Bibby Sterilin types 122, Glasgow, UK), and measured in the sample chamber for 120 s. The photon counts of separated minerals were recorded (7).

Thermoluminescence (TL) measurement and evaluation

TL measurement was carried out using a TL reader (Harshaw 3500, Wermelskirchen, Germany) with temperatures 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 and was measured in units of nano coulombs (nC). After the first glow curve was measured, the discs with the minerals were subsequently re-irradiated using a Co-60 gamma rays with a normalizing dose of 1 and 5 kGy. The TL intensity was measured again after the re-irradiation step (second glow curve). The glow curve ratios 1 (G1) (first glow curve of unirradiated sample/second glow curve of irradiated sample at 1 and 5 kGy) and 2 (G2, G3, G4) (first glow curve per irradiation dose (G2=1 kGy, G3=5 kGy, G4=10 kGy)/second glow curve of irradiated spices at 1 and 5 kGy) were then determined. TL measurements of all samples were repeated three times (27).

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 shape of the glow curves is needed to decide whether the sample has been irradiated or not, since the shapes of first glow curve showed in higher temperature region than those of the second glow curve (27).

Statistical analysis

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

RESULTS AND DISCUSSION

The changes in photon counts of the mineral extracted from irradiated sesame and perilla seeds

Table 1 shows the changes in photon counts of the mineral of irradiated sesame seeds measured by PPSL for 60 s and regression expressions and coefficients between irradiation dose and photon counts. The photon counts of the mineral of unirradiated and irradiated sesame seeds at 1, 5, and 10 mg exhibited an increase from 8,517±990, 27,276±4,380, and 44,723±1,349 respectively, in the unirradiated control to 1,563,196±584,968, 4,582,919±188,672, and 8,257,229 ±444,891 in the mineral of irradiated sesame seeds at 5 kGy, and slightly decreased except for 5 mg to 885,793±263,913, 5,306,349±748,930, and 7,801,860±743,488 at 10 kGy.

The photon counts of mineral extracted from the irradiated perilla and sesame seeds measured by PPSL for 120 s are shown in Table 2. The photon counts of the mineral measured for 120 s exhibited an increase with increasing irradiation dose and mineral content, and this trend was similar for all groups. Also, the photon counts of all the irradiated sesame and perilla seed samples measured for 120 s were higher than those measured for 60 s. In all the samples, the photon counts of minerals of all irradiated sesame and perilla seed samples were higher than those of the unirradiated ones (Fig. 1). Hence, the authors believe that the detection of irradiated perilla and sesame seeds is possible by PPSL in both 60 s and 120 s measurement time, and PPSL can be proposed as a method to detect the irradiation treatment of perilla and sesame seeds.

Similar results for PPSL have been reported. Sanderson et al. (1) reported that the photon count of intestinal grits and irradiated bown shrimp samples was higher than unirradiated samples. Chung et al. (9) reported that PSL values of irradiated white ginseng powder were negative for unirradiated samples but were positive for irradiated ones. Hwang et al. (8) also reported that the photon counts of irradiated pepper powder, dried herbs, fresh shrimp, potato, soybean, dried fig, chestnut, dried squid, and dried cod increased with dosage. Our results above agree with those reported by other researchers (1,6,7).

TL intensity, glow curve ratio, and maximum TL temperature of the mineral separated from irradiated sesame and perilla seeds

TL intensity, glow curve ratio, and maximum TL temperature of the mineral separated from irradiated sesame and perilla seeds are shown in Table 3. As shown in Table 3, TL intensities of minerals separated from irradiated sesame and perilla seeds increased from 159.713±18.801 and 182.670 ± 12.992 in the unirradiated sample to 2215.544±171.691 and 5706.698±372.545 at 10 kGy, respectively (Fig. 2). Regression expressions and coefficients of minerals separated from irradiated sesame and perilla seeds were $y = -34.337x^2$

Table 1. The changes of accumulated photon counts of minerals separated from irradiated sesame and perilla seeds measured by PPSL during the 60 s, and regression expressions and coefficients between irradiation dose and photon counts (Unit : photon counts)

Materials	Mineral contents (mg)	Irradiation dose (kGy)				Regression expressions and coefficients	
		Control$^{1)}$	1	5	10	From control to 5 kGy	From control to 10 kGy
Sesame seed	1	^{A}8,517^{a} ±990	^{A}1,145,408^{b} ±155,222	^{A}1,563,196^{c} ±584,968	^{A}885,793^{b} ±263,913	y = 251939x+401828 R^2 = 0.6864	y = -45268x^2+509101x+290260 R^2 = 0.8044
	5	^{B}27,276^{a} ±4,380	^{AB}1,960,782^{b} ±434,701	^{B}4,582,919^{c} ±188,672	^{B}4,673,016^{c} ±553,548	y = 838102x+514118 R^2 = 0.9405	y = -89954x^2+1E+06x+316736 R^2 = 0.9823
	10	^{C}44,723^{a} ±1,349	^{C}2,628,150^{b} ±695,775	^{C}8,257,229^{c} ±444,891	^{A}7,801,860^{c} ±743,488	y = 2E+06x+492774 R^2 = 0.9851	y = -173597x^2+2E+06x+152417 R^2 = 0.9992
Perilla seed	1	^{I}3,022^{a} ±1,732	^{I}589,704^{b} ±44,483	^{I}1,692,484^{d} ±181,997	^{I}1,117,834^{c} ±108,075	y = 320122x+121492 R^2 = 0.9750	y = -45348x^2+561687x+32465 R^2 = 0.9982
	5	$^{I\,II}$18,053^{a} ±10,195	^{II}2,357,409^{b} ±232,837	^{II}5,672,295^{d} ±349,104	^{II}3,660,351^{c} ±311,625	y = 1E+06x+593527 R^2 = 0.9461	y = -153900x^2+2E+06x+277079 R^2 = 0.9873
	10	^{III}33,383^{a} ±12,545	^{III}3,532,351^{b} ±266,902	^{III}8,641,921^{d} ±449,464	^{III}6,087,570^{c} ±291,582	y = 2E+06x+879689 R^2 = 0.9498	y = -224113x^2+3E+06x+418184 R^2 = 0.9883

$^{1)}$Control = unirradiated sample

$^{2)a-d}$Means with the same superscripts in each row are not significantly different among group by Duncan's multiple range test in one way ANOVA ($p<0.05$).

$^{3)A-C}$Means with the same superscripts in each column are not significantly different among group by Duncan's multiple range test in one way ANOVA ($p<0.05$).

$^{4)I-III}$Means with the same superscripts in each column are not significantly different among group by Duncan's multiple range test in one way ANOVA ($p<0.05$) Mean value±Standard Deviation for 3 measurements.

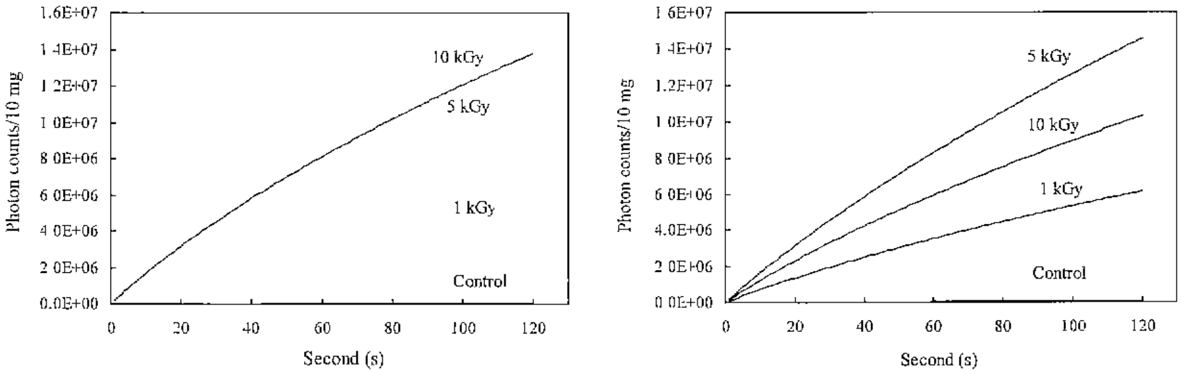
Table 2. The changes of accumulated photon counts of minerals separated from irradiated sesame and perilla seeds measured by PPSL during the 120 s, and regression expressions and coefficients between irradiation doses and photon counts (Unit : photon counts)

Materials	Mineral contents (mg)	Irradiation dose (kGy)				Regression expressions and coefficients	
		0	1	5	10	From control to 5 kGy	From control to 10 kGy
Sesame seed	1	^{A}13,981^{a} ±2,615	^{A}1,562,917^{b} ±292,933	^{A}2,840,431^{c} ±340,867	^{A}1,439,354^{b} ±426,445	y = 495030x+482381 R^2 = 0.8563	y = -85175x^2+963076x+294874 R^2 = 0.9372
	5	^{B}48,382^{a} ±7,530	^{B}2,981,029^{b} ±128,539	^{B}7,712,995^{c} ±275,931	^{B}8,291,921^{d} ±2,048,235	y = 1E+06x+714910 R^2 = 0.9610	y = -142520x^2+2E+06x+410836 R^2 = 0.9921
	10	^{C}80,113^{a} ±3,198	^{C}4,208,246^{b} ±649,142	^{C}14,067,998^{c} ±909,627	^{C}13,775,553^{c} ±2,262,194	y = 3E+06x+713695 R^2 = 0.9898	y = -285789x^2+4E+06x+161959 R^2 = 0 9999
Perilla seed	1	^{I}5,056 ±2,849	^{I}997,768^{b} ±81,578	^{I}2,842,091^{d} ±285,502	^{I}1,929,297^{c} ±272,661	y = 537029x+207580 R^2 = 0.9740	y = -75118x^2+937544x+59589 R^2 = 0.9979
	5	$^{I\,II}$43,935^{a} ±38,083	^{II}3,700,036^{b} ±1,126,836	^{II}9,784,131^{d} ±581,619	^{II}6,380,419^{c} ±540,396	y = 2E+06x+857288 R^2 = 0.9641	y = -263514x^2+3E+06x+329646 R^2 = 0.9949
	10	^{III}58,827^{a} ±21,457	^{III}6,167,958^{b} ±441,745	^{III}14,538,001^{d} ±1,241,836	^{III}10,331,114^{c} ±950,029	y = 3E+06x+2E+06 R^2 = 0.9418	y = -375384x^2+5E+06x+806655 R^2 = 0.9844

$^{1)-4)}$Refer to the legend in Table 1.

+540.27x+237.92 (R^2 = 0.9937) and y = -90.121x^2+1423.2x +456.5 (R^2 = 0.9937), and showed high correlation between irradiation doses and TL intensities in a multinomial expression (Fig. 3). Generally, the TL intensities of minerals separated from irradiated samples were higher than those of unirradiated samples (10,19-24). Schreiber et al. (18) reported that TL emission occurs when the excited electrons return to the original level at a certain temperature.

A comparison of the glow curve ratio as well as an analysis of glow curve shapes were preferred for identifying irradiated and unirradiated samples in several studies (4,19,21). Table 3 shows the TL intensities of the second glow curve and the glow curve ratios for irradiated sesame and perilla seeds. As the glow curve ratios (G1) of the unirradiated sesame and perilla seeds were less than 0.1, they were classified as unirradiated. The glow curve ratios (G4) for irradiated ones were greater than 0.5 and the typical glow shapes for irradiated samples were observed. Thus, all irradiated samples could be classified correctly. Hammerton and Banos (28) reported that all samples were clearly identified by re-irradiation as either unirradiated or irradiated and the TL ratios (glow curve ratio) varied between 0.0039 and 0.19 for unirradiated samples, and between 0.79 and 2.4 for samples irradiated with 5 kGy. Moreover, the shape of the glow curves could classify all irra-



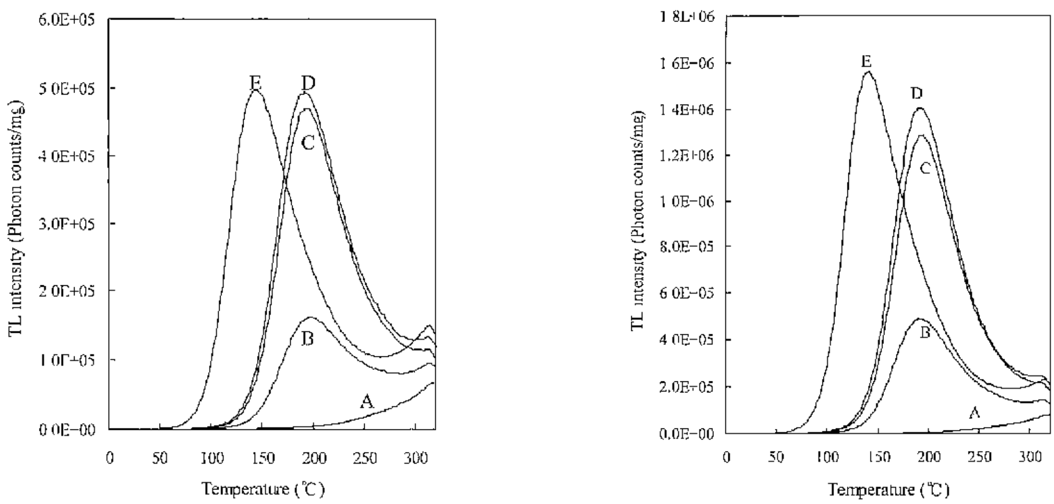
A) Mineral seperated from irradiated sesame seeds B) Mineral seperated from irradiated perilla seeds

Fig. 1. PPSL curves of minerals seperated from irradiated sesame and perilla seeds at various doses.

Table 3. The first, second glow curves and glow curve ratios of irradiated sesame and perilla seeds (Unit : nano coulombs (nC/mg))

Materials	Glow curves			Irradiation dose (kGy) Control[1)]	1	5	10
Sesame seed	First glow curve			159±18[a]	852±150[b]	2,041±447[c]	2,215±171[c]
	Second glow curve			G1[2)]	G2[3)]	G3[4)]	G4[5)]
	Glow curve ratio	1 kGy	2,422±661	0.0659	0.3519	0.8429	0.9147
		5 kGy	4,376±845	0.0364	0.1947	0.4665	0.5062
Perilla seed	First glow curve			182±12[a]	2,169±274[b]	5,182±875[c]	5,706±372[c]
	Second glow curve			G1	G2	G3	G4
	Glow curve ratio	1 kGy	6,602±970	0.0276	0.3286	0.7849	0.8643
		5 kGy	11,085±2215	0.0164	0.1957	0.4675	0.5148

[1)]Control = unirradiated sample
[2)]G1 = first glow curve of unirradiated sample/second glow curve of re-irradiated sample at 1 or 5 kGy
[3)]G2 = first glow curve of irradiated sample at 1 kGy/second glow curve of re-irradiated sample at 1 or 5 kGy
[4)]G3 = first glow curve of irradiated sample at 5 kGy/second glow curve of re-irradiated sample at 1 or 5 kGy
[5)]G4 = first glow curve of irradiated sample at 10 kGy/second glow curve of re-irradiated sample at 1 or 5 kGy
[a-c]Means with the same superscripts in each row are not significantly different among group by Duncan's multiple range test in one way ANOVA ($p<0.05$).



A) Mineral seperated from irradiated sesame seeds B) Mineral seperated from irradiated perilla seeds

Fig. 2. TL first and second glow curves of mineral seperated from irradiated sesame and perilla seeds at various doses.
A) Unirradiated sample B) 1 kGy C) 5 kGy D) 10 kGy E) Second glow curve reirradiated at 1 kGy

Table 4. The maximum TL temperatures of the first glow curves and the second glow curves for irradiated sesame and perilla seeds (Unit : °C)

Sample	First glow curves				Second glow curves	
	Control[1)]	1 kGy	5 kGy	10 kGy	1 kGy	5 kGy
Sesame seed	320.00 ± 0.00^c	197.78 ± 5.66^b	195.07 ± 3.93^b	191.89 ± 2.08^b	145.65 ± 4.78^a	147.01 ± 2.83^a
Perilla seed	320.00 ± 0.00^c	195.52 ± 1.36^b	195.52 ± 3.59^b	193.71 ± 1.57^b	142.93 ± 3.42^a	140.21 ± 0.79^a

[1)]Control = unirradiated sample

[a-c]Means with the same superscripts in each row are not significantly different among group by Duncan's multiple range test in one way ANOVA ($p<0.05$).

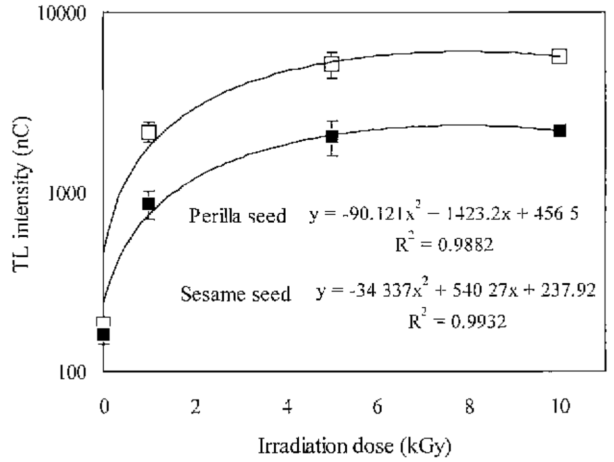


Fig. 3. Regression curves of TL intensities of mineral sepeiated from irradiated sesame and perilla seeds at various doses. □, Perilla seed; ■, Sesame seed.

diated sesame and perilla seeds correctly. Interpretation of the shape of the glow curves should be studied to determine whether a sample has been irradiated or not, if the glow curve ratio is between 0.1 and 0.5 (27).

Maximum TL temperatures of the first and second glow curves of minerals separated from irradiated sesame and perilla seeds are presented in Table 4. Maximum TL temperatures were $191.89 \pm 2.08 \sim 197.78 \pm 5.66$ and $193.71 \pm 1.57 \sim 195.52 \pm 1.36$°C, respectively, and those of second glow curves (re-irradiated 1 kGy) were 145.65 ± 4.78 and 142.93 ± 3.42°C, respectively. Maximum TL temperatures of all irradiated sesame and perilla seeds were below 200°C, within the 150~250°C temperature interval recommended by DIN EN 1788 (27), whereas unirradiated samples did not exhibit maximum TL temperatures and similar results were obtained by many other researchers (4,11,20). Based on the above results, all irradiated samples were correctly classified as irradiated.

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