# Detection of Irradiated Korean Wheat Flour by Viscosity and Pulsed Photostimulated Luminescence (PPSL) Methods

Sang-Duk Yi1, Kyu-Seob Chang2 and Man-Jin Oh2t

<sup>1</sup>Nonghyup Korea Insam Research Institute, Nonghyup Koreainsam Co., LTD., Chungbuk 386-811, Korea <sup>2</sup>Department of Food Science and Technology, Chungnam National University, Daejeon 305-764, Korea

#### **Abstract**

This study was carried out to establish methods for irradiation detection of irradiation in Korean wheat flour by pulsed photostimulated luminescence (PPSL) and viscometric methods. The photon counts of the irradiated Korean wheat flour measured by PPSL immediately after irradiation increased with increasing irradiation dose. The photon counts in the irradiated Korean wheat flour almost disappeared with lapse of time after storage in normal room conditions, but irradiation detection was still possible after 6 months in darkroom conditions. All irradiated samples indicated a decrease in viscosity with increasing stirring speeds (rpm) and irradiation doses. Irradiation at 1 kGy significantly decreased the viscosity. Consequently, these results suggest that the detection of irradiated Korean wheat powder is possible by both viscometric and PPSL methods.

Key words: detection methods, viscosity, PPSL, gamma irradiation, Korean wheat

### INTRODUCTION

Irradiation can be used for disinfecting as well as for the reduction of microbial contamination thereby reducing health hazards which might be caused by pathogenic microorganism and prevention of sprouting or delay ripening of fruit and vegetables (1-3). Detection techniques of irradiated foods have been required to control international trade, confirm correct labeling, avoid multiple irradiations and control the absorbed dose of irradiated foods (4-6). Recently, the detection of irradiated foods is required by many consumers. Therefore, the methods to detect irradiated foods are a useful means to check compliance with labeling regulations (7,8).

Pulsed photostimulated luminescence (PPSL), which uses light rather than heat to stimulate the release of trapped charge from carriers of irradiation energy, has been utilized for irradiated foodstuffs as a detection method which is designed to allow direct measurements for rapid screening purposes without the need for sample preparation (9,10).

Viscosity measurement has also been proposed as a method to detect irradiation treatment of foods containing high amounts of starch and have been carried out for some foods (11-15).

Generally, starch is composed of amylose and amylopectin. These two major component are degraded by

gamma irradiation, resulting in a decrease in viscosity (16-26). Therefore, the objectives of this work were to establish viscometric and PPSL methods for detecting irradiated Korean wheat flour, which have not yet been examined in previous studies, and to describe the results for changes in viscosity and PPSL.

#### MATERIALS AND METHODS

#### Materials and irradiation

The Korean wheat flour used for this study was purchased from Dusan Co. (Icheon, Gyeonggi, Korea). The samples were packed in polyethylene bags and irradiated with 1, 2, 3, 5, 7 and 10 kGy using a Co-60 irradiator (AECL, IR-79, Ontario, Canada) at 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.

## Measurement of pulsed photostimulated luminescence (PPSL)

The PPSL system is composed of a control unit, sample chamber, and detector head assembly; the control unit contains a stimulation source which is comprised of an array of infra-red light emitting diodes which are pulsed symmetrically on and off for equal periods. PPSL is detected by a bialkali cathode photomultiplier tube

<sup>†</sup>Corresponding author. E-mail: ohmj@cnu.ac.kr Phone: +82-42-821-6728. Fax: +82-42-821-6728 operating in photon counting mode. Optical filtering is used to define both the stimulation and detection wavebands. The samples (5 g) were introduced in 50 mm diameter disposable petri dishes (Bibby Sterilin type 122), with no other preparation, and measured in the sample chamber for 60 and 120 s. The photon counts of the samples were recorded (9). Samples stored at normal room conditions were stored in laboratory conditions with sunlight and an electric light. Samples stored in dark room conditions were stored in a dry oven (K.M.C-1203P3, Vision Scientific Co., LTD, Seoul, Korea) at room temperature.

#### Measurement of viscosity

Viscosity was measured according to Hayashi et al.'s (20-22) method with a slight modification. The Korean wheat flour was dissolved in distilled water to make an 8% aqueous solution to which 2.14 mL of 33% NaOH was added. The samples were mixed thoroughly for 30 sec. The glass bottle was heated for about 30 min in an autoclave (100°C). The glass bottle was left in an incubator (30°C) for 3 hr to maintain a uniform temperature. Viscosity was determined using a Brookfield DV-III rotation viscometer (Brookfield Engeineering Laboratories Inc., USA) equipped with an RV3 spindle at 30°C, and measured at 60, 120 and 180 rpm.

#### Statistical analysis

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

experiments were repeated three times.

#### RESULTS AND DISCUSSION

#### Changes in photon counts

The changes in photon count of the irradiated Korean wheat flour measured by PPSL are shown in Table 1. The photon counts from the irradiated Korean wheat flour measured for 60 and 120 s exhibited higher than from non-irradiated. Also, the photon counts from samples measured for 120 s were higher than those measured for 60 s. In all samples, the photon counts of the irradiated Korean wheat flour samples were higher than those of the non-irradiated samples when measured immediately after irradiation (control). Hence, the authors believe that irradiation detection of Korean wheat flour is possible by PPSL in both 60 s and 120 s measurement time. Differences in photon count according to storage conditions (normal room and darkroom) were clearly observed. In darkroom conditions, the photon counts of irradiated Korean wheat flour were observed after 6 months, but the photon count after one month in normal room conditions was almost undetectable (Table 2). As irradiated samples showed photon counts higher than those of non-irradiated samples in darkroom conditions, in this condition, irradiation detection was still possible after 6 months. Therefore, we think that PPSL has potential as a method for detecting the irradiation treatment of Korean wheat flour. Similar results for PPSL have been reported by other investigators. Sanderson et al. (26)

**Table 1.** Changes in accumulated photon counts of non-irradiated and irradiated Korean wheat flour according to storage conditions duration of storage (Unit: photon counts)

Storage time		Measurement	Irradiation dose (kGy)					
	ondition	time (sec)	0	1	5	10		
0 month		60 120	$260 \pm 85^{1)c2)} \\ 232 \pm 121^{c}$	$15,253 \pm 4,449^{\mathrm{b}} \\ 25,335 \pm 7,630^{\mathrm{b}}$	$47,046 \pm 10,007^{a}$ $67,953 \pm 9,518^{a}$	$45,597 \pm 6,045^{a}$ $64,795 \pm 12,234^{a}$		
, ,	Normal room	60 120	$271 \pm 28^{c}$ $417 \pm 82^{c}$	$2,996 \pm 2,373^{a}$ $3,983 \pm 1,603^{a}$	$1,390 \pm 631^{a}$ $2,139 \pm 1,259^{a}$	$1,781 \pm 712^{a}$ $2,997 \pm 1,057^{a}$		
1 month	Dark room	60 120	336±36° 515±234°	14,291 ± 5,551 <sup>b</sup> 23,544 ± 9,389 <sup>b</sup>	$\begin{array}{cccccccccccccccccccccccccccccccccccc$	$39,471 \pm 4,676^{a}$ $54,407 \pm 6,712^{a}$		
2	Normal room	60 120	536±233 <sup>b</sup> 468±245 <sup>b</sup>	$960 \pm 134^{a}$ $1,510 \pm 284^{a}$		627 ± 47 <sup>b</sup> 979 ± 374 <sup>b</sup>		
3 months	Dark room	60 120	$306 \pm 33^{\circ} \\ 294 \pm 164^{\circ}$	$10,515 \pm 1,994 \\ 17,385 \pm 3,326^{b}$		$26,173 \pm 3,719^{a} 34,881 \pm 5,287^{a}$		
6 o tho	Normal room	60 120	353±230 <sup>b</sup> 311±55 <sup>c</sup>	$478 \pm 104^{\rm b} \\ 656 \pm 144^{\rm a}$		996±527 <sup>a</sup> 1,556±991 <sup>a</sup>		
6 months	Dark room	60 120	$323 \pm 139^{c}$ $370 \pm 159^{c}$	$7,905 \pm 750^{\mathrm{b}} \\ 13,239 \pm 1,240^{\mathrm{b}}$		$21,244 \pm 3,050^{a} 30,881 \pm 5,287^{a}$		

<sup>&</sup>lt;sup>1)</sup>Mean value ± standard deviation for 3 measurements.

<sup>&</sup>lt;sup>2)</sup>Means with the same superscripts in each row are not significantly different by Duncan's multiple range test and one way ANOVA (p < 0.05).

and storage conditions	1			 (Unit: %)
Storage time	Measurement	Irradiati	on dose (kGy)	

Storage time & condition		Measurement	Irradiation dose (kGy)					
		time (sec)	0	1	5	10		
0		60	NC <sup>1)</sup>	0	0	0		
0 month		120	NC	0	0	0		
	Normal room	60 .	NC	80.3	97.1	96.1		
1		120	NC	84.3	96.9	95.4		
1 month	Dark room	60	NC	6.3	32.1	13.4		
		120	NC	7.1	26.7	16.0		
	Normal room	60	NC	93.7	98.8	98.6		
2		120	NC	94.0	98.5	97.7		
3 months		60	NC	31.1	63.9	42.6		
	Dark room	120	NC	31.3	1 5 0 0 0 0 0 80.3 97.1 84.3 96.9 6.3 32.1 7.1 26.7 93.7 98.8 94.0 98.5 31.1 63.9	23.5		
	Normal room	60	NC	97.1	98.3	97.8		
6 mantha		120	NC	97.4	97.5	96.6		
6 months	Dark room	60	NC	48.2	61.9	53.4		
		120	NC	47.7	50.5	32.2		

<sup>1)</sup>Sample not calculated.

reported that the photon counts of intestinal grits and all irradiated shrimp were higher than non-irradiated ones. In principle, a PPSL emission occurs when the excited electrons return to the original level due to light (2,3). Consequently, these results suggest that detection of irradiated Korean wheat flour is possible by both PPSL methods.

#### Changes of viscosity

Fig. 1, 2 and 3 show the viscosity of irradiated Korean wheat powder at different dose levels and stirring speeds (rpms). Viscosity was reduced as dose levels and stirring speeds increased. Viscosity according to increasing irradiation doses was dramatically reduced from control with just 1 kGy of irradiation, and slowly reduced even further

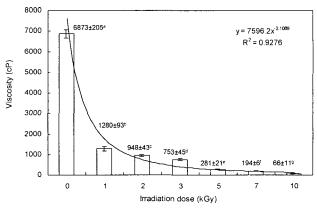
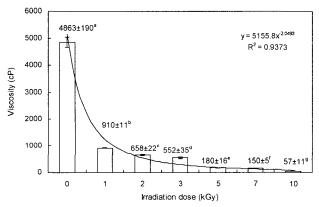


Fig. 1. Changes in viscosity and regression curves between irradiation dose and viscosity of irradiated Korean wheat flour measured at 60 rpm. Mean value  $\pm$  standard deviation for 3 measurements. <sup>a-d</sup>Means with the same superscripts in each row are not significantly different by Duncan's multiple range test and one way ANOVA (p < 0.05).



**Fig. 2.** Change in viscosity and regression curves between irradiation dose and viscosity of irradiated Korean wheat flour measured at 120 rpm. Mean value  $\pm$  standard deviation for 3 measurements. <sup>a-d</sup> Means with the same superscripts in each row are not significantly different by Duncan's multiple range test and one way ANOVA (p < 0.05).

with doses of 2 kGy to 10 kGy (Table 3). The viscosities of non-irradiated Korean wheat flour were higher than in the irradiated flour in all samples. Therefore, it seems that if the viscosity of an unknown Korean wheat flour sample as measured by a viscometer is below or above that of an non-irradiated flour, we can distinguish whether it is irradiated or not. Based on these results, irradiation detection in Korean wheat flour is possible by a viscometric method, and this can be proposed as a detection method of irradiated Korean wheat flour.

Similar results for decreases in starch viscosity by irradiation have been reported. MacArthur and D'Appolonia (11) reported that reduced viscosity in irradiated starch was due to the degradation and uncoiling of starch chains, as well as the breaking of hydrogen bonds within

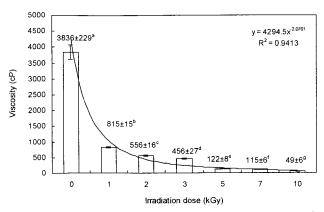


Fig. 3. Change of viscosity and regression curves between irradiation dose and viscosity of irradiated Korean wheat powder measured at 180 rpm. Mean value  $\pm$  standard deviation for 3 measurements. <sup>a-d</sup>Means with the same superscripts in each row are not significantly different among group by Duncan's multiple range test and one way ANOVA (p < 0.05).

**Table 3.** Viscosity decay rate of irradiated and non-irradiated Korean wheat powder (Unit: %)

*****		Ir	radiatio	n dose	(kGy)		
rpm	0	1	2	3	5	7	10
60	0	81.3	86.2	89.0	95.9	97.2	99.0
120	0	81.2	86.6	88.6	96.3	96.9	98.8
180	0	78.7	85.5	88.1	96.8	97.0	98.7

the molecule. Roushdi et al. (12) also reported that decreased viscosity for gamma irradiated corn starch was due to the decreased starch chain length.

The reduction of viscosity in irradiated starch was caused by the free radicals created by gamma irradiation. Free radicals are responsible for molecular changes such as the uncoiling of starch chains and fragmentation by the breaking of hydrogen bonds in the starch molecules. These changes may affect the physical and rheological properties of starch, thus decreasing the viscosity (13-15). The coefficients of variation  $(0.92 \sim 0.94)$  were very high between irradiation dose and viscosity (Fig. 1, 2 and 3). Hence, these results suggest that viscosity measurement a reliable and useful method to detect irradiation and that the PPSL method is necessary for rapid screening irradiated Korean wheat powder.

## **REFERENCES**

- 1. Delincée H. 1998. Detection of food treated with ionizing radiation. *Trends Food Sci Technol* 9: 73-82.
- Sanderson DCW, Carmichael LA, Spencer JQ, Naylor JD. 1996. Luminescence detection of shellfish. In *Detection methods for irradiated foods*. McMurray CH, Stewart EM, Gray R, Pearce J, eds. The Royal Society of Chemistry, UK. p 139-148.
- 3. Sanderson DCW, Carmichael LA, Naylor JD. 1996. Recent advances in thermoluminescence and photostimulated

- luminescence detection methods for irradiated foods. In *Detection methods for irradiated foods.* McMurray CH, Stewart EM, Gray R, Pearce J, eds. The Royal Society of Chemistry, UK. p 124-138.
- Yi SD, Yang JS. 2001. Properties of pulsed photostimulated luminescence and thermoluminescence of gamma-irradiated shrimp-taste seasoning powder. Food Sci Biotechnol 10: 408-413.
- Yi SD, Woo SH, Yang JS. 2001. Detection of pulsed photostimulated luminescence signals emitted by infrared stimulation of irradiated spices during storage under two conditions. J Food Sci Nutr 6: 152-157.
- Chung HW, Delincée H, Kwon JH. 2000. Photostimulated luminescence-thermoluminescence application to detection of irradiated white ginseng powder. *Korean J Food Sci Technol* 32: 265-270.
- Hwang KT, Uhm TB, Wagner U, Schreiber GA. 1998. Application of photostimulated luminescence to detection of irradiated foods. Korean J Food Sci Technol 30: 498-501
- Schreiber GA. 1996. Thermoluminescence and photostimulated luminescence techniques to indentify irradiated foods. In *Detection methods for irradiated foods*. McMurray CH, Stewart EM, Gray R, Pearce J, eds. The Royal Society of Chemistry, Cambridge, UK. p 121-123.
- European Committee for standard. 1997. Detection of irradiated food using photostimulated luminescence. English version of prEN 13751.
- Yi SD, Yang JS. 2001. Influence of sample form, storage conditions and periods on accumulated pulsed photostimulated luminescence signals of irradiated Korean sesame and perilla seeds. *J Food Sci Nutr* 6: 216-223.
- 11. MacArthur LA, D'Appolonia BL. 1984. Gamma radiation of wheat. II. Effects of low-dosage radiations on starch properties. *Cereal Chem* 61: 321-326.
- Roushdi M, Harras A, El-meligi A, Bassim M. 1983. Effect of high doses of gamma rays on corn grains. Starke 35: 15-21.
- 13. Nene SP, Vakil UK, Sreenivasan A. 1975. Effect of gamma radiation on physio-chemical characteristics of red gram (*Cajanus cajan*) starch. *J Food Sci* 40: 943-947.
- 14. Sokhey AS, Hanna MA. 1993. Properties of irradiated starches. *Food Structure* 12: 397-410.
- Yi SD, Chang KS, Yang JS. 2000. Detection of irradiated cereals by viscosity measurement. *J Food Sci Nutr* 5: 93-99.
- Yi SD, Chang KS, Yang JS. 2000. Identification of irradiated potato, sweet potato and corn starches with viscometric method. Food Sci Biotechnol 9: 57-62.
- 17. Yi SD, Oh MJ, Yang JS. 2000. Detection for irradiated cereals by maximum viscosity in amylograph. *Food Sci Biotechnol* 9: 73-76.
- Farkas J, Koncz A, Sharif MM. 1990. Identification of irradiated dry ingredients on the basis of starch damage. *Radiat Phys Chem* 35: 324-328.
- Farkas J, Sharif MM, Koncz A. 1990. Detection of some irradiated spices on the basis of radiation induced damage of starch. *Radiat Phys Chem* 36: 621-627.
- Hayashi T, Todoriki S, Kohyama K. 1994. Irradiation effects on pepper starch viscosity. J Food Sci 59: 118-120.
- Hayashi T, Todoriki S. 1996. Detection of irradiated peppers by viscosity measurement at extremely high pH. Radiat Phys Chem 48: 101-104.
- 22. Hayashi T, Kawashima K. 1982. The effect of gamma irradiation on the sucrose content in sweet potato roots and

- potato tubers. Agric Biol Chem 46: 1475-1479.
- 23. Heide L, Nrnberger E, Bögl KW. 1990. Investigations on the detection of irradiated food measuring the viscosity of suspended spices and dried vegetables. *Radiat Phys Chem* 36: 613-619.
- 24. Schreiber GA, Leffke A, Mager M, Helle N, Bögl KW. 1994. Viscosity of alkaline suspensions of ground black and white pepper samples: An indication or an identification of high dose radiation treatment? *Radiat Phys Chem*
- 44: 467-472.
- 25. Sharif MM, Farkas J. 1993. Analytical studies into radiation-induced starch damage in black and white pepers. *Radiat Phys Chem* 42: 383-386.
- Sanderson DCW, Carmichael LA, Riain SN, Naylor J, Spencer JQ. 1994. Luminescence studies to identify irradiated food. Food Science and Technology Today 8: 93-96

(Received February 2, 2005; Accepted March 15, 2005)