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Trial to Identify Irradiated Corn Powder by Viscometric and Pulsed Photostimulated Luminescence (PPSL) Methods

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ABSTRACT – A study was performed to establish detection methods by viscometric and pulsed photostimulated luminescence (PPSL) methods for irradiated corn powder. Viscosity was determined using a Brookfield DV- rotation viscometer at 30°C and operated at 30, 60, 90, 120, 150, 180, and 210 rpm. All irradiated samples showed a decrease in viscosity with increasing stirring speeds (rpm) and irradiation doses. Treatments at 1~3 kGy significantly decreased the viscosity. The photon counts of irradiated corn powder were measured by PPSL immediately after irradiation and exhibited an increase with increasing irradiation dose. The photon counts of irradiated corn powder almost disappeared with lapse of time in room conditions, but detection of irradiation was still possible after one month at darkroom conditions. Consequently, these results suggest that the detection of irradiated corn powder is possible by both viscometric and PPSL methods.

Key words □ detection methods, viscosity, PPSL, gamma irradiation, corn powder

Corn powder is utilized as a major ingredient of flaking grits and snack grits, which are processed into many corn-based food products, such as corn flakes, breakfast cereals, corn chips, extruded snacks, tortillas, and many other snack foods. It can be contaminated by microorganisms such as *Fusarium moniliforme, F. subglutinans, F. proliferatum, and F. graminearum* producing food poisoning fumonisins B1 (FB1), B2 (FB2), and B3 (FB3) associated with several animal diseases, including pulmonary edema in swine, liver cancer in rats, acute congestive heart failure in baboons and monkeys, and human cancer.[1)] Ionizing radiation can be used for disinfecting as well as for the reduction of microbial contamination, reducing health hazards which might be caused by pathogenic microorganism and preventing sprout or delaying maturity of fruit and vegetables.[2)] Hence, to sterilize corn powder, gamma irradiation has become increasingly important. However, additional means of identifying irradiated food has been particularly requested by consumer organizations, and in recent years an enormous interest in detection methods for irradiated food has been developed, which may reflect an increased awareness about the benefits of food irradiation because correct and comprehensive information about food irradiation and irradiated food must reach consumers in order to enable them to make decisions based on well-founded reasons.[3)]

Previously, viscometric detection of irradiated foods has been carried out for peppers, spices,[4-14)] starches and cereals[25-28)] and it has been proposed as a method to detect irradiation treatment of foods containing high amounts of starch. Pulsed photostimulated luminescence (PPSL), which uses light rather than heat to stimulate the release of trapped charge carriers following irradiation, has been tried for irradiated foodstuffs as a detection method which is designed to allow direct measurements for rapid screening purposes without sample preparation.[15-18)] Therefore, the objectives of this work based on this background were to establish viscometric and PPSL methods for detecting irradiated corn powder.

Materials and Methods

Materials and irradiation

The corn powder used for this study was purchased from Dusan Co. (Ich'on, Kyonggi, Korea). The contents of starch as a dried matter and moisture of corn powder were 0.59 ± 0.08 g/g and 7.00 ± 0.47%, respectively. The samples were packed in polyethylene bags and irradiated at 1, 2, 3, 5, 7, 10, and 15 kGy using a Co-60 irradiator (AECL, IR-79, Ontario, Canada) with a dose rate of 10 kGy/h at

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Korea Atomic Energy Research Institute. To measure total absorbed dose ceric-cerous dosimeter was used. Samples were divided into two groups of storage conditions, room condition and dark condition at room temperature.

Determination of starch and moisture contents

Moisture content was measured by the AOAC method.[19] Starch content was determined according to Hayashi and Kawashima's method[20] with a slight modification.

Viscosity measurement

Viscosity was measured according to the method described by Hayashi et al[6-10] with a slight modification. The corn powder was placed in a glass bottle and an 8% aqueous solution was prepared. After adding 2.14 mL of 33% NaOH, 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 the uniform temperature. Viscosity was determined using a spindle RV 3 of the Brookfield DV-III rotation viscometer (Brookfield Engeineering Laboratories Inc., USA) at 30°C, and operated at 30, 60, 90, 120, 150, 180, and 210 rpm.

Determination of parameters

Identification parameters A, B, and C were calculated as follows: parameter A=viscosity per irradiation dose/moisture content, parameter B=parameter A per irradiation dose/amount of starch in a 1 g sample, and parameter C =parameter B per irradiation dose/control of parameter B.

Measurement of pulsed photostimulated luminescence (PPSL)

The PPSL system is composed of control unit, sample chamber, and detector head assembly. The control unit contains a stimulation source which comprised 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 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 samples were recorded.[15-18]

Results and Discussion

Viscosity changes

Table 1 shows the viscosity of irradiated corn powder at different dose levels and stirring speeds (rpms). Viscosity reduced as dose levels and stirring speeds increased. In the control and all irradiation doses, viscosity according to increased stirring speeds reduced from the control to 1 kGy group, but showed a contrary trend from 2 kGy to 15 kGy. The viscosities of unirradiated corn powder were higher than those of irradiated ones (Fig. 1). Therefore, it seems that if the viscosity of the unknown corn powder sample as measured by a viscometer is below or above that of an unirradiated corn powder sample, we can distinguish whether it has been irradiated or not. Based on the results, the detection of irradiated corn powder was possible by a viscometric method, and this can be proposed as a method for detecting of irradiated corn powder.

Similar results on the decrease in starch viscosity by irradiation have been reported. MacArthur and D'Appolonia[21] reported that reduced viscosity in irradiated starch was due to the degradation and uncoiling of starch chains, as well

Table 1. The changes of apparent viscosity of irradiated corn powders at various doses and stirring speeds

Rpm	Irradiation dose (kGy)							
	0	1	2	3	5	7	10	15
30	298.9 ± 13.4[1)]	192.3 ± 13.4	117.2 ± 5.8	92.2 ± 5.0	70.2 ± 3.0	51.2 ± 2.0	46.9 ± 5.6	31.6 ± 1.7
60	270.0 ± 14.6	181.7 ± 9.3	109.8 ± 34.8	88.9 ± 3.4	70.2 ± 4.5	52.5 ± 3.7	49.6 ± 4.7	38.8 ± 0.9
90	257.2 ± 13.9	169.7 ± 0.7	110.0 ± 5.1	90.3 ± 2.8	72.8 ± 3.5	59.9 ± 1.1	53.7 ± 3.6	42.8 ± 0.6
120	249.7 ± 11.7	166.1 ± 0.51	111.2 ± 6.3	94.2 ± 2.2	77.8 ± 3.2	63.7 ± 0.9	58.9 ± 3.1	48.0 ± 0.5
150	241.6 ± 8.5	164.3 ± 0.6	112.2 ± 8.0	98.9 ± 1.7	82.9 ± 3.2	70.3 ± 1.7	64.1 ± 2.8	52.5 ± 0.4
180	237.2 ± 6.77	165.0 ± 0.7	118.2 ± 6.3	103.5 ± 1.4	86.1 ± 2.8	74.8 ± 0.7	68.5 ± 3.2	56.4 ± 0.7
210	234.3 ± 6.6	166.5 ± 1.02	121.8 ± 5.7	106.8 ± 1.2	88.7 ± 2.4	79.7 ± 1.1	72.8 ± 2.1	60.2 ± 0.8

[1)]Means ± standard deviation for 3 measurements

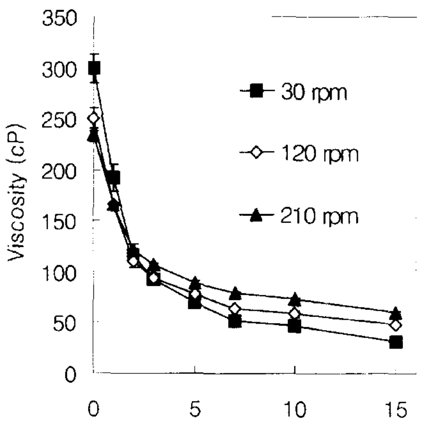

Fig. 1. The changes of apparent viscosity of irradiated corn powders measured at various doses and stirring speeds.

as the breaking of hydrogen bonds within the molecule. Roushdi et al.[22] also reported that decreased viscosity for gamma irradiated corn starch was due to the decreased starch chain length. Nene et al.[23] found that irradiated (10 kGy) red gram flour exhibited a very low maximal gelatinization viscosity (350 B.U.) compared to the control (860 B.U.).

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.[24] Hence, the reason for the reduction in viscosity in corn powder was guessed similar to that of starch explained by other researchers.[21-24]

Parameter values and regression expressions between viscosity and irradiation dose

The parameter values derived from maximum viscosity and the viscosity of corn powder were listed in Table 2. A normalized parameter would be better for detecting irradiation treatment than the viscosity value itself, because moisture and starch contents of corn powder show influences on viscosity, which is affected by experimental equip-

Table 2. Parameter values derived from apparent viscosity of irradiated and unirradiated corn powders at various doses and stirring speeds

Rpm	Parameter values	Irradiation Dose (kGy)							
		0	1	2	3	5	7	10	15
30	PA[1)]	42.95	27.63	16.83	13.25	10.09	7.36	6.73	4.54
	PB[2)]	72.80	46.83	28.53	22.46	17.10	12.47	11.41	7.69
	PC[3)]	1.0000	0.6434	0.3921	0.3085	0.2349	0.1713	0.1569	0.1057
60	PA	38.79	26.11	15.78	12.77	10.09	7.54	7.13	5.57
	PB	65.75	44.25	26.75	21.64	17.10	12.78	12.08	9.44
	PC	1.0000	0.6730	0.4067	0.3293	0.2600	0.1944	0.1837	0.1419
90	PA	36.95	24.38	15.80	12.97	10.46	8.61	7.72	6.15
	PB	62.63	41.32	26.78	21.98	17.73	14.59	13.08	10.42
	PC	1.0000	0.6598	0.4277	0.3511	0.2830	0.2329	0.2088	0.1664
120	PA	35.88	23.86	15.98	13.53	11.18	9.15	8.46	6.90
	PB	60.81	40.44	27.08	22.93	18.95	15.51	14.34	11.69
	PC	1.0000	0.6652	0.4453	0.3773	0.3116	0.2551	0.2359	0.1922
150	PA	34.71	23.61	16.12	14.21	11.91	10.10	9.21	7.54
	PB	58.83	40.02	27.32	24.08	20.19	17.12	15.61	12.78
	PC	1.0000	0.6800	0.4644	0.4094	0.3431	0.2910	0.2653	0.2173
180	PA	34.08	23.71	16.98	14.87	12.37	10.75	9.84	8.10
	PB	57.76	40.19	28.78	25.20	20.97	18.22	16.68	13.73
	PC	1.0000	0.6956	0.4983	0.4363	0.3630	0.3153	0.2888	0.2378
210	PA	33.66	23.92	17.50	15.34	12.74	11.45	10.46	8.65
	PB	57.05	40.54	29.66	26.00	21.59	19.41	17.73	14.66
	PC	1.0000	0.7106	0.5198	0.4558	0.3786	0.3402	0.3107	0.2569

1)Parameter A 2)Parameter B 3)Parameter C ments and techniques of researchers. Therefore, we believe that the apparent viscosity divided by moisture (parameter A), starch content (parameter B), and irradiated samples divided by the control of parameter B (parameter C) will provide detection values to reduce the variation of the viscosity values among the irradiated corn powder samples. Among them, parameter C is the best for detecting irradiated corn powder due to its inherent viscosity. Regression equations and coefficients of viscosity according to irradiation doses of corn powder were listed in Table 3. The coefficients (0.980.99) were very high between irradiation dose and viscosity.

Photon counts under storage conditions

The changes in photon counts of the irradiated corn powder measured by PPSL are shown in Table 4 and Fig. 1. The photon counts of corn powder measured for 60 and 120 s exhibited an increase with increasing irradiation dose. Also, the photon counts of the corn powder samples measured for 120 s were higher than those measured for 60 s. In both samples measured immediately after irradiation (control) and after 1 month in darkroom condition

Table 3. Regression equations and coefficients derived from the viscosity of irradiated and unirradiated corn powders

Rpm	Regression expressions	Coefficients
30	$y = 13.424x^2-111.55x+294.87$	$R^2 = 0.9957$
60	$y = 11.788x^2-98.167x+268.13$	$R^2 = 0.9935$
90	$y = 10.898x^2-90.186x+253.48$	$R^2 = 0.9926$
120	$y = 10.328x^2-84.752x+245.7$	$R^2 = 0.9906$
150	$y = 9.5739x^2-78.368x+237.72$	$R^2 = 0.9884$
180	$y = 8.6048x^2-72.081x+233.48$	$R^2 = 0.9898$
210	$y = 7.9861x^2-67.993x+230.94$	$R^2 = 0.9909$

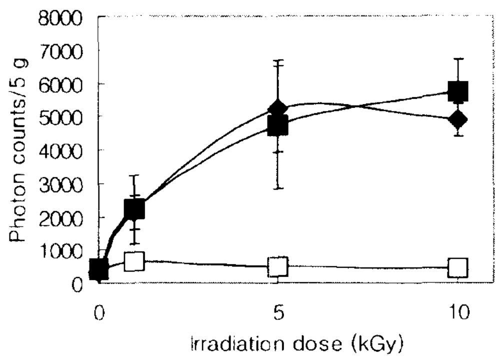

Fig. 2. The curves of accumulated photon counts of unirradiated and irradiated corn powders according to storage conditions.

for 60 s and 120 s measurement time, as the photon count of the corn powder samples were higher than those of the unirradiated ones, the authors believe that detection of the corn powder is possible by PPSL in both measurement time. The differences in photon counts according to storage conditions (room and darkroom) were clearly observed. In darkroom conditions, significant changes in the photon counts of corn powder was insignificant, but the photon count after one month at room conditions was not almost observed. In darkroom conditions, as irradiated samples showed photon counts higher than those of unirradiated samples, detection of irradiation was still possible after one month. Therefore, we think that PPSL can be proposed as a method for detecting the irradiation treatment of corn powder. Similar results for PPSL have been reported. Sanderson et al.[15] reported that the photon counts of intestinal grits and all irradiated Bown Shrimp were higher than those

Table 4. The changes of accumulated photon counts of unirradiated and irradiated corn powder according to storage conditions and periods (Unit: Photon counts)

Storage conditions & periods		Measurement time (second)	Irradiation dose (kGy) 0	1	5	10
Control[1]		60	168.33 ± 124.52	1780.33 ± 392.27	4158.00 ± 1154.88	5220.67 ± 1600.31
		120	477.00 ± 167.11	2215.33 ± 1034.77	4738.33 ± 1927.62	5744.33 ± 971.77
Darkroom condition	1 month	60	125.00 ± 21.93	1500.33 ± 391.09	3920.67 ± 1692.69	2735.33 ± 580.29
		120	333.00 ± 214.41	2132.33 ± 508.43	5202.00 ± 1297.92	4882.00 ± 493.65
Room condition	1 month	60	402.00 ± 143.52	460.67 ± 69.95	321.33 ± 99.28	268.33 ± 47.69
		120	415.00 ± 125.87	653.67 ± 202.96	497.00 ± 112.60	431.67 ± 22.12

[1]Control = Photon counts measured immediately after irradiation
[2]Means ± standard deviation for 3 measurements.

of unirradiated ones. Our results agreed with those reported by Sanderson et al.[15] In principle, a PPSL emission occurs when the excited electrons return to the original level due to light. Consequently, these results suggest that irradiated corn powder is possibly detected by both PPSL and viscometric methods.

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국문요약

본 연구는 점도 측정법과 광여기발광법을 이용하여 방사선 조사된 옥수수분말의 검지를 시도하였으며, 이전의 연구에서 수행되지 않은 다양한 rpm에서의 검지 가능성과 저장기간에 따른 광여기발광의 변화를 검토하였다. 실험에 사용된 옥수수분말은 polyethylene bag 으로 포장하였고, Co-60 감마선 조사시설을 이용하여 1, 2, 3, 5, 7, 10 및 15 kGy로 조사하였다. 점도측정 시료는 증류수를 가하여 현탁하고 알카리화 한 후에 RV 3 spindle이 장착된 Brookfield DV-III rotation viscometer를 이용하여 30℃에서 30초 동안 30, 60, 90, 120, 150, 180 및 210 rpm 에서 점도를 측정하였다. 광여기발광 측정은 시료(5 g)의 전처리 없이 직접 광여기발광기에 넣은 후에 측정하였다. 모든 시료의 점도는 조사선량과 stirring speeds (rpm)가 증가할수록 감소하는 경향을 보였으며, 조사선량과 점도간의 높은 상관성을 보여 주었다. 방사선 조사된 옥수수 분말의 광여기발광은 조사선량이 증가할수록 증가하는 경향을 보였고, 1개월 후에도 암실조건에서의 광여기발광은 조사직후의 시험구에 비하여 감소하지 않았다. 이상의 결과를 종합하여 볼 때 점도 측정법과 광여기발광법은 방사선 조사된 옥수수분말의 조사여부를 확인할 수 있는 검지법으로의 가능성을 확인할 수 있었다.

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