# Application of Viscometric Method for the Detection of Irradiated Black and White Pepper

Sang-Duk Yi, Kyu-Seob Chang\* and Jae-Seung Yang†

Detection Lab. of Irradiated Food, Korea Atomic Energy Research Institute Taejon 305-353, Korea \*Dept. of Food Science and Technology, Chungnam National University, Taejon 305-764, Korea

## 방사선 조사된 후추가루의 검지를 위한 점도법의 적용

이상덕 · 장규섭\* · 양재승

한국원자력연구소 조사식품의 검지기술 개발실, \*충남대학교 식품공학과

ABSTRACT – A study was carried out to establish a detection method for irradiated black and white pepper. Samples were packed in polyethylene bags and irradiated with 2.5, 5, 7.5, 10 and 15 kGy using a Co-60 irradiator. The samples were suspended in water, and alkalized with sodium hydroxide solution. Apparent viscosity was determined after heat gelatinization using a Brookfield DV-III rotation viscometer at 30°C with 30, 60, 90, 120, 150, 180, and 210 rpm. Means and standard deviations of the viscosities of all samples decreased by increasing the stirring speeds. The viscosities increased in all samples by increasing the concentration. Regression expressions and coefficients of viscosity which decreased with increasing irradiation dose of 10% and 13% black pepper, and 7% and 10% white pepper were 0.9531 (y=-131.29x+1,769.0), 0.9725 (y=-351.33x+4,036.0), 0.9731 (y=2,208.0e<sup>-0.3546x</sup>), and 0.9959 (y=5,116.0e<sup>-0.2887x</sup>), respectively, at 120 rpm. This trend was similar for all stirring speeds. These results suggest that the detection of irradiated black and white pepper at various doses is possible by the viscometric method.

Key words 
Detection methods, Viscosity, Gamma irradiation, Pepper

#### INTRODUCTION

Pepper is one of the major food items treated with ionizing radiation because pepper in its natural state is highly contaminated with molds, yeasts and bacteria, and is very vulnerable to insect infestation. For the increasing demand for decontaminated spices, until recently, fumigation which used chemical agents such as ethylene oxide and propylene oxide has been the main method used for the decontamination of spices, though it has many problems. Heating the spices at 38~60°C for 6~8 hr can decrease the volatile compounds and the ethylene oxide can form toxic, flammable and explosive mixtures with air. Hence in many countries ionizing radiation has become an economically viable alternative

as a decontamination process and is increasingly being used for the microbial decontamination of dry food ingredients, such as spices and herbs. 4) Pepper is irradiated in 26 countries as of 1995<sup>5)</sup> and the trade of irradiated pepper will increase, since irradiation does not adversely affect the quality and safety of the product. Many health authorities and consumer organizations demand unequivocal tests for the identification of irradiated foods. The existence of simple and effective tests would be highly desirable to supplement the control of trade in irradiated foods.<sup>7)</sup> Recently, detection studies for irradiated foods have been widely performed with physical, chemical, and biological methods. 8,9) Among these detection techniques, viscometric detection studies were mainly carried out for dried vegetables and spices using a viscometer and the measurement of viscosity has been proposed as a method to detect the irradiation treatment of these food

<sup>&</sup>lt;sup>†</sup>Author to whom correspondence should be addressed.

products. 10-17) Starch is degraded by ionizing radiation, resulting in a decrease in viscosity. 18-26) The viscosities of black and white pepper, which contain large amounts of starch,2) are also reduced by irradiation, so that viscosity measurement has been proposed as a method to detect irradiation treatment of black and white pepper. Moreover, it is a useful detection technique because the viscosity measurements widely carried out at public and private laboratories dealing with foods, simple and inexpensive. 6 Another advantage is that viscosity changes are quite stable on storage. 2,3) On the basis of this background, the aim of this study was to clarify the effects of viscosity conditions on the viscosity value, to establish parameters for detecting irradiated pepper which is independent of viscosity measuring conditions, and to add new data in this field, such as the influence of various rpms and regression expressions and coefficients not yet examined in viscometric detection using a viscometer.

#### MATERIALS AND METHODS

#### Materials and irradiation

Black and white pepper harvested in Malaysia was purchased from a supplier. The moisture contents of black and white pepper were  $13.15\pm0.50\%$  and  $13.40\pm0.70\%$ , and starch contents of dried black and white pepper were  $0.46~\rm g\pm0.01~\rm g/g$  and  $0.67~\rm g\pm0.05~\rm g/g$ , respectively. The moisture content was measured by the AOAC method. The starch content was determined according to Hayashi and Kawashima's method using the enzyme reaction of glucose oxidase and peroxidase. The samples were packed in polyethylene bags and irradiated with 2.5, 5, 7.5, 10 and 15 kGy using a Co-60 irradiator (AECL, Canada) with a dose rate 166.6 Gy/min at the Korea Atomic Energy Research Institute. A ceric-cerous dosimeter was used to measure the exact total absorbed doses.

#### Measuring method of apparent viscosity

Viscosity was measured according to Hayashi et al's method<sup>2,6,13-15)</sup> with a slight modification. Black and white pepper were placed in glass bottles to prepare an aqueous solution (10% and 13% black pepper, 7% and 10% white pepper). After adding 2.14 ml of 33% NaOH, the samples

were mixed thoroughly for 30 sec. The glass bottles were heated for about 30 min in a 95°C water bath (occasional stirring). The glass bottles were left in an incubator (30°C) for 3 hr to maintain uniform temperature. The viscosity was determined using the spindle RV 6 of a Brookfield DV-III rotation viscometer (Brookfield Engeineering Laboratories Inc., USA) at 30°C and measured at 30, 60, 90, 120, 150, 180, and 210 rpm.

## Calculation of parameter originated from apparent viscosity

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/starch amount in 1 g of sample, and parameter C=parameter B per irradiation dose/the control of parameter B. Parameter C removed the variation affected by viscosity was listed as a main parameter.

#### Statistical analysis

Significant difference was determined by using Duncan's multiple range test (p < 0.05) in a one-way ANOVA, and regression expressions and coefficients were determined through the regression analysis of the SPSS (Statistical Package for Social Science) version 7.5. All experiments were repeated three times.

#### RESULTS AND DISCUSSION

## Changes of viscosity according to concentration and various stirring speeds for irradiated black pepper

To study the changes of viscosity according to concentration and various stirring speeds for irradiated black and white pepper, a study was performed with samples of 7% and 13% concentration prepared for this study, and samples of 10% concentration used in previous studies. The viscosities of irradiated black pepper are shown in Tables 1 and 2. The viscosities of unirradiated black pepper (control) prepared with 10% concentration at 30, 60, 90, 120, 150, 180, and 210 rpm were measured at  $4,471.3\pm73.6$ ,  $2,836.7\pm59.1$ ,  $2,145.3\pm11.6$ ,  $1,769.7\pm4.6$ ,  $1,523.0\pm10.0$ ,  $1,350.3\pm6.0$  and  $1,223.7\pm12.7$  cP, respectively. The viscosities of the samples irradiated at 15 kGy were measured at  $411.1\pm38.4$ ,  $300.0\pm0.0$ ,  $255.6\pm5.3$ ,  $230.5\pm4.8$ ,  $219.9\pm11.5$ ,  $205.6\pm3.8$  and  $188.9\pm$ 

Irradiation Dose (kGy) rpm Control1) 2.5 10 15 7.5 $\overline{^{\text{A3})}}4,471.3\pm73.6^{\text{a2}}$ 30  $^{\text{A}}4,021.7 \pm 19.6^{\text{t}}$  $^{\text{A}}2,881.3 \pm 183.5^{\circ}$  $^{\text{A}}1,533.3 \pm 100.0^{\circ}$ ^771.2 ± 67.1°  $^{\text{A}}411.1 \pm 38.4^{\text{f}}$ 60  $^{B}2,836.7 \pm 59.1^{a}$  $^{\mathrm{B}}2,575.7 \pm 8.1^{\mathrm{b}}$  $^{\rm B}$ 1,865.3  $\pm$  87.1  $^{\rm c}$  $^{\rm B}$ 1.029.0  $\pm$  34.3 $^{\rm d}$  $^{\mathrm{B}}551.87 \pm 33.5^{\mathrm{e}}$  $^{\mathrm{B}}300.0 \pm 0.0^{\mathrm{f}}$ 90  $^{\rm c}$ 2,145.3  $\pm$  11.6 $^{\rm a}$  $^{\rm c}$ 1,989.0  $\pm$  11.0 $^{\rm b}$  $^{c}1.459.0 \pm 39.2^{c}$  $^{\text{C}}829.6 \pm 33.9^{\text{d}}$  $^{\text{C}}438.2 \pm 27.8^{\text{e}}$  $^{\rm c}255.6 \pm 5.3^{\rm f}$ 120  $^{\mathrm{D}}1,769.7\pm4.6^{\mathrm{a}}$  $^{\mathrm{D}}1,655.0\pm18.0^{\mathrm{b}}$  $^{\mathrm{D}}1,223.3\pm25.2^{\circ}$  $^{\mathrm{D}}734.4 \pm 25.1^{\mathrm{d}}$  $^{\text{CD}}384.5\pm16.8^{\text{e}}$  $^{\text{CD}}230.5 \pm 4.8^{\text{f}}$  $^{\rm E}$ 1,523.0 ± 10.0 $^{\rm a}$  $^{\text{DE}}655.8 \pm 27.0^{\text{d}}$  $^{ ext{DE}}349.1 \pm 16.8^{\circ}$  $^{\rm E}$ 1,450.7  $\pm$  10.7  $^{\rm b}$  $^{\mathrm{E}}1.074.7\pm13.7^{\circ}$ D219.9 ± 11.5f 150 180  $^{\rm F}$ 1,350.3  $\pm$  6.0 $^{\rm a}$  $^{\text{F}}1,296.7 \pm 16.9^{\text{b}}$  $^{\text{EF}}957.0 \pm 13.9^{\circ}$  $^{\text{EF}}598.8 \pm 25.2^{\text{d}}$ EF319.7 ± 8.3°  $^{DE}205.6 \pm 3.8^{f}$  $^{G}$ 1,223.7  $\pm$  12.7 $^{a}$ 210  $^{G}1,172.3\pm10.9^{b}$  $^{F}879.4 \pm 2.8^{c}$  $^{\text{F}}543.1 \pm 14.3^{\text{d}}$  $^{\text{F}}290.5 \pm 8.3^{\text{e}}$  $^{E}188.9 \pm 2.8^{f}$ 

Table 1. Viscosities of black pepper prepared with a 10% concentration at various doses and rpms (Unit: centipoise (cP))

Table 2. Viscosities of black pepper prepared with a 13% concentration at various doses and rpms (Unit: centipoise (cP))

rpm	Irradiation Dose (kGy)							
	Control <sup>1)</sup>	2.5	5	7.5	10	15		
30	$^{3)A}9,744.0 \pm 952.9^{a2)}$	<sup>A</sup> 8,488.7 ± 269.6 <sup>b</sup>	<sup>A</sup> 5,499.3 ± 284.8 <sup>c</sup>	$^{\text{A}}2,577.9 \pm 146.6^{\text{d}}$	<sup>A</sup> 1,770.0 ± 34.0 <sup>e</sup>	<sup>A</sup> 1,422.3 ± 38.7 <sup>e</sup>		
60	$^{\mathrm{B}}6,201.0\pm500.0^{\mathrm{a}}$	$^{\mathrm{B}}$ 5,298.0 $\pm$ 132.1 $^{\mathrm{b}}$	$^{\mathrm{B}}3,518.0\pm201.8^{\mathrm{c}}$	$^{\mathrm{B}}1,622.4\pm91.9^{\mathrm{d}}$	$^{\mathrm{B}}1,196.7\pm67.2^{\mathrm{e}}$	$^{\mathrm{B}}961.1\pm19.2^{\mathrm{e}}$		
90	$^{\rm c}$ 4,911.0 $\pm$ 326.0 $^{\rm a}$	$^{\mathrm{c}}4,009.3 \pm 88.1^{\mathrm{b}}$	$^{\rm c}$ 2,762.3 $\pm$ 145.0 $^{\rm c}$	$^{\rm c}$ 1,285.9 $\pm$ 17.2 $^{\rm d}$	$^{\rm c}898.3 \pm 53.6^{\rm e}$	$^{\rm c}$ 759.3 $\pm$ 6.4 $^{\rm e}$		
120	$^{\mathrm{D}}4,036.7\pm219.7^{\mathrm{a}}$	$^{\mathrm{D}}3,319.\pm399.6^{\mathrm{b}}$	$^{\mathrm{D}}2,322.0\pm134.5^{\mathrm{c}}$	$^{\mathrm{D}}1,024.5\pm37.4^{\mathrm{d}}$	$^{\mathrm{D}}743.3 \pm 20.8^{\mathrm{e}}$	$^{\mathrm{D}}646.7 \pm 5.8^{\mathrm{e}}$		
150	$^{ ext{DE}}3,445.0\pm160.6^{ ext{a}}$	$^{\mathrm{E}}2,793.7\pm65.2^{\mathrm{b}}$	$^{\mathrm{E}}$ 2,013.3 $\pm$ 115.5 $^{\mathrm{c}}$	$^{\rm E}852.6\pm83.6^{\rm d}$	$^{\mathrm{E}}644.8\pm12.0^{\mathrm{e}}$	$^{\mathrm{E}}576.9 \pm 8.5^{\mathrm{e}}$		
180	$^{\mathrm{E}}3,070.3\pm122.2^{\mathrm{a}}$	$^{\mathrm{F}}$ 2,455.3 $\pm$ 83.9 $^{\mathrm{b}}$	$^{\mathrm{EF}}1,814.0\pm135.0^{\mathrm{c}}$	$^{\mathrm{EF}}783.3\pm70.7^{\mathrm{d}}$	$^{\mathrm{E}}585.0\pm17.2^{\mathrm{e}}$	$^{\text{F}}520.4 \pm 6.4^{\text{e}}$		
210	$^{\mathrm{E}}2,753.7\pm86.2^{\mathrm{a}}$	$^{\text{F}}2,235.0 \pm 23.5^{\text{b}}$	$^{\text{F}}1,655.0 \pm 141.6^{\circ}$	$^{\text{F}}660.7 \pm 90.7^{\text{d}}$	$^{\text{F}}509.6 \pm 21.9^{\text{e}}$	$^{6}479.3\pm5.5^{\circ}$		

Refer to the legend in Table 1. Means  $\pm$  Standard Deviation for 3 measurements

2.8 cP, respectively. The viscosities decreased according to increasing irradiation dose in all groups.

The viscosity of unirradiated black pepper (control) and irradiated black pepper prepared with 10% concentration on 2.5, 5, 7.5, 10, and 15 kGy dropped from  $4,471.3\pm73.6,\ 4,021.7\pm19.6,\ 2,881.3\pm183.5,\ 1,533.3\pm$ 100.0,  $771.2 \pm 67.1$ , and  $11.1 \pm 38.4$  cP, respectively, at 30 rpm to  $1,223.7 \pm 12.7$ ,  $1,172.3 \pm 10.9$ ,  $879.4 \pm 2.8$ ,  $543.1\pm14.3$ ,  $290.5\pm8.3$ , and  $188.9\pm2.8$  cP, respectively, at 210 rpm (Table 1). The viscosities showed a reduction with increasing doses. The viscosity of unirradiated black pepper (control) and irradiated black pepper prepared with 13% concentration showed a reduction with increasing doses and stirring speeds. This tendency was similar for the viscosities of black pepper prepared with 10% concentration (Table 2). Significant variations in the viscosity of black pepper prepared with 10% concentration and 13% concentration showed differences between irradiation dose and same trend between increasing stirring speeds (p<0.05).

Similar results for black pepper have been reported. De

Alwis and Grandison<sup>3)</sup> reported that irradiated black pepper showed a marked reduction in viscosity and higher irradiation doses gave rise to higher reductions in apparent viscosity. Most of the above results agreed with those reported by many other researchers, as well as de Alwis and Grandison.<sup>1-4)</sup>

Generally, black pepper has a high amount starch and that with higher starch contents showed higher viscosity values.<sup>2)</sup> Hence, the reason that the viscosity values of black pepper prepared with 13% concentration were higher than those of black pepper prepared with 10% concentration seems to be caused by the difference starch content of the sample.

### Changes of viscosity according to concentration and various stirring speeds for irradiated white pepper

Table 3 shows the viscosities of unirradiated and irradiated white pepper prepared with 7% concentration. The viscosities of unirradiated white pepper prepared with 7% concentration at 30, 60, 90, 120, 150, 180, and 210 rpm were measured at  $4,611.0\pm183.7,\ 3,199.0\pm$ 

<sup>&</sup>lt;sup>1)</sup>Control: unirradiated sample.

<sup>&</sup>lt;sup>2) a-h</sup> Means with the same superscripts in each row are not significantly different (p  $\langle 0.05 \rangle$ . (n=3)

<sup>&</sup>lt;sup>3) A-G</sup> Means with the same superscripts in each column are not significantly different (p  $\langle 0.05 \rangle$ ). (n=3)Means  $\pm$  Standard Deviation for 3 measurements

Irradiation Dose (kGy) rpm Control<sup>1)</sup> 10 15 2.5 7.5 30  $^{6)A}4,611.0\pm183.7^{a2)}$  $^{\text{A}}$ 1,667.0 ± 100.0  $^{\text{b}}$  $^{A}400.0 \pm 0.0^{\circ}$  $^{\text{A}}200.0 \pm 0.0^{\text{d}}$  $^{\text{A}}88.9 \pm 19.2^{\text{d}}$  $^{\text{A}}66.7 \pm 0.0^{\text{d}}$  $^{\rm B}$ 1,238.7  $\pm$  58.7  $^{\rm b}$  $^{AB}77.7 \pm 9.6^{\circ}$ 60  $^{\mathrm{B}}3,199.0\pm67.0^{\mathrm{a}}$  $^{\mathrm{B}}350.0 \pm 0.0^{\mathrm{c}}$  $^{\rm B}172.2\pm9.6^{\rm d}$  $^{\mathrm{B}}50.0 \pm 0.0^{\mathrm{e}}$  $^{\text{C}}2.577.3 \pm 61.8^{\text{a}}$  $^{\text{C}}1.044.3 \pm 50.9^{\text{b}}$  $^{\rm C}149.7 \pm 5.6^{\rm d}$  $^{AB}77.8 \pm 0.0^{e}$  $^{\text{C}}44.4 \pm 6.7^{\text{e}}$ 90  $^{\rm c}311.1 \pm 0.0^{\rm c}$ 120  $^{\mathrm{D}}2.208.7 \pm 50.0^{\mathrm{a}}$ D922.2 ± 37.6b  $^{\text{D}}288.9 \pm 4.8^{\circ}$  $^{\text{C}}154.6 \pm 4.2^{\text{d}}$  $^{AB}72.2 \pm 4.8^{e}$  $^{\text{C}}45.8 \pm 4.2^{\text{e}}$  $^{\text{DE}}840.0 \pm 33.3^{\text{b}}$  $^{\mathrm{B}}68.9\pm7.7^{\mathrm{c}}$  $^{E}1,972.3 \pm 51.6^{a}$  $^{E}268.9 \pm 3.8^{\circ}$  $^{\text{CD}}144.4 \pm 7.7^{\text{d}}$  $^{\rm c}43.7 \pm 3.4^{\rm e}$ 150  $^{\mathrm{EF}}$ 774.1  $\pm$  28.0 $^{\mathrm{b}}$  $^{DE}138.5 \pm 5.6^{d}$  $^{\text{F}}251.9 \pm 3.2^{\circ}$  $^{\mathrm{B}}67.8 \pm 5.9^{\mathrm{e}}$  $^{\rm C}42.6 \pm 3.2^{\rm e}$ 180  $^{\text{F}}1,793.3 \pm 45.3^{\circ}$  $^{\rm F}$ 1,653.0  $\pm$  35.6 $^{\rm a}$  $^{G}234.9 \pm 2.8^{\circ}$  $^{\text{F}}720.7 \pm 27.1^{\text{b}}$  $^{\rm E}132.2\pm5.1^{\rm d}$  $^{\mathrm{B}}63.5 \pm 2.8^{\mathrm{e}}$  $^{\rm c}42.9\pm0.0^{\rm e}$ 210

Table 3. Viscosities of white pepper prepared with a 7% concentration at various doses and rpms(Unit: centipoise (cP))

67.0, 2,577.3 $\pm$ 61.8, 2,208.7 $\pm$ 50.0, 1,972.3 $\pm$ 51.6, 1,793.3  $\pm$ 45.3 and 1,653.0 $\pm$ 35.6 cP, respectively. The viscosities of the sample irradiated at 15 kGy were measured at 66.7 $\pm$ 0.0, 50.0 $\pm$ 0.0, 44.4 $\pm$ 6.7, 45.8 $\pm$ 4.2, 43.7 $\pm$ 3.4, 42.6 $\pm$ 3.2 and 42.9 $\pm$ 0.0 cP, respectively. The viscosities showed a marked reduction with increasing doses. This trend was similar for all stirring speeds. Increasing stirring speeds gave rise to a reduction in viscosity in all groups.

The changes of viscosities for unirradiated and irradiated white pepper prepared with 10% concentration are given in Table 4. As shown in Table 4, the viscosities of white pepper prepared with 10% concentration measured at 30, 60, 90, 120, 150, 180, and 210 rpm dropped from  $10,816.3\pm256.6$ ,  $7,003.3\pm62.0$ ,  $5,985.3\pm25.4$ ,  $5,166.7\pm44.4$ ,  $4,874.7\pm203.8$ ,  $4,188.3\pm141.8$  and  $3,951.3\pm10.1$  cP, respectively, in the unirradiated control to  $234.3\pm33.4$ ,  $205.6\pm25.4$ ,  $191.8\pm22.8$ ,  $177.1\pm17.1$ ,  $166.7\pm20.0$ ,  $154.4\pm16.8$  and  $149.9\pm19.5$  cP, respectively, for the samples irradiated at 15 kGy and decreased according to increasing stirring speeds. The viscosities of white pepper prepared with 10% concentration were higher than those of white pepper prepared with 7% concentration. As the concentration of the sample

increased, the viscosity increased with a concentration dependent relationship in white pepper. This tendency was similar for all stirring speeds. Significant variations in the viscosity of white pepper prepared with 7% concentration and 10% concentration showed clear differences both with increasing stirring speeds and irradiation doses (p<0.05).

Similar results for white pepper have been reported. Hayashi et al.<sup>14)</sup> reported that the viscosity of white pepper suspensions decreased with doses for all white pepper samples, and many other researchers also reported similar results. The results in our study were consistent with those reported by many other researchers. <sup>9-17)</sup>

In principle, the other researchers<sup>20-26)</sup> explained that the reduction of viscosity in irradiated starch seems to be caused by the free radicals created by gamma irradiation. Increasing dosages of gamma irradiation creates increasing intensities of free radicals in carbohydrates, which are responsible for molecular changes such as the uncoiling of starch chains and fragmentation by the breaking of hydrogen bonds of the starch molecules. These changes may affect the physical and rheological properties of starch and decrease viscosity.

Based on the above papers, the reason for the reduction

Table 4. Viscosities of white pepper prepared with a 10% concentration at various doses and rpms centipoise (cP))

	Irradiation Dose (kGy)							
rpm	Control <sup>1)</sup>	2.5	5	7.5	10	15		
30	$^{3)A}10,816.3 \pm 256.6^{a2)}$	<sup>A</sup> 7,477.7 ± 533.9 <sup>b</sup>	$^{\text{A}}2,014.7 \pm 16.8^{\circ}$	^877.8 ± 279.6 <sup>d</sup>	$^{\text{A}}416.7 \pm 104.1^{\text{de}}$	^234.333.4°		
60	$^{\mathrm{B}}$ 7,003.3 $\pm$ 62.0 $^{\mathrm{a}}$	$^{\mathrm{B}}4,859.0\pm324.9^{\mathrm{b}}$	$^{\mathrm{B}}1,575.7\pm8.1^{\mathrm{c}}$	$^{\mathrm{AB}}700.0 \pm 204.8^{\mathrm{d}}$	$^{\mathrm{AB}}338.7 \pm 78.6^{\mathrm{e}}$	AB 205.625.4°		
90	$^{\rm c}$ 5,985.3 $\pm$ 25.4 $^{\rm a}$	$^{\text{c}}$ 3,564.7 $\pm$ 666.5 $^{\text{b}}$	$^{\mathrm{c}}$ 1,382.0 $\pm$ 66.9 $^{\mathrm{c}}$	$^{AB}637.0 \pm 200.1^{d}$	$^{\mathrm{AB}}299.6\pm69.2^{\mathrm{d}}$	BC 191.822.8d		
120	$^{\mathrm{D}}5,166.7\pm44.4^{\mathrm{a}}$	$^{\text{CD}}$ 3,011.3 $\pm$ 514.8 $^{\text{b}}$	$^{\mathrm{D}}1,191.7\pm55.0^{\mathrm{c}}$	$^{\mathrm{AB}}575.0\pm175.0^{\mathrm{d}}$	$^{\mathrm{B}}278.1 \pm 61.6^{\mathrm{d}}$	<sup>BC</sup> 177.117.1 <sup>d</sup>		
150	$^{\mathrm{D}}4,874.7\pm203.8^{\mathrm{a}}$	$^{\text{CD}}2,690.3 \pm 645.8^{\text{b}}$	$^{\mathrm{E}}$ 1,064.3 $\pm$ 23.5°	$^{AB}528.9 \pm 144.9^{d}$	$^{\mathrm{B}}263.5\pm55.2^{\mathrm{d}}$	BC 166.720.0d		
180	$^{\mathrm{E}}4,188.3\pm141.8^{\mathrm{a}}$	$^{\mathrm{D}}2,329.0\pm407.9^{\mathrm{b}}$	$^{\rm F}$ 1,009.0 $\pm$ 8.5 $^{\rm c}$	$^{\mathrm{B}}503.7 \pm 142.2^{\mathrm{d}}$	$^{\mathrm{B}}246.3 \pm 50.4^{\mathrm{de}}$	<sup>c</sup> 154.416.8 <sup>e</sup>		
210	$^{\mathrm{E}}3,951.3\pm10.1^{\mathrm{a}}$	$^{\mathrm{D}}2,100.7\pm250.0^{\mathrm{b}}$	$^{\text{F}}967.2 \pm 21.1^{\circ}$	$^{\rm B}476.2\pm133.3^{\rm d}$	$^{\mathrm{B}}238.1 \pm 49.5^{\mathrm{e}}$	<sup>c</sup> 149.919.5 <sup>e</sup>		

Refer to the legend in Table 1. Means ± Standard Deviation for 3 measurements

<sup>1)-4)</sup> Refer to the legend in Table 1. Means ± Standard Deviation for 3 measurements

Table 5. Parameter C of irradiated black pepper and white pepper at various doses and rpms

		Irradiation Dose (kGy)						
	rpm	Control <sup>1)</sup>	2.5	5	7.5	10	15	
	30	1.0000	0.8994	0.6444	0.3429	0.1725	0.0919	
$A^{2)}$	60	1.0000	0.9080	0.6576	0.3627	0.1946	0.1057	
	90	1.0000	0.9271	0.6801	0.3867	0.2042	0.1191	
	120	1.0000	0.9352	0.6913	0.4150	0.2172	0.1303	
	150	1.0000	0.9525	0.7057	0.4306	0.2292	0.1443	
	180	1.0000	0.9603	0.7087	0.4434	0.2368	0.1522	
	210	1.0000	0.9580	0.7186	0.4438	0.2373	0.1544	
	30	1.0000	0.8712	0.5643	0.2646	0.1816	0.1460	
	60	1.0000	0.8543	0.5673	0.2616	0.1930	0.1550	
	90	1.0000	0.8163	0.5625	0.2618	0.1829	0.1546	
$\mathbf{B}^{3)}$	120	1.0000	0.8223	0.5752	0.2538	0.1841	0.1602	
	150	1.0000	0.8109	0.5843	0.2475	0.1872	0.1675	
	180	1.0000	0.7997	0.5909	0.2551	0.1906	0.1695	
	210	1.0000	0.8119	0.6010	0.2399	0.1850	0.1741	
	30	1.0000	0.3615	0.0867	0.0433	0.0193	0.0145	
	60	1.0000	0.3872	0.1094	0.0538	0.0243	0.0156	
	90	1.0000	0.4052	0.1207	0.0581	0.0302	0.0172	
$C^{4)}$	120	1.0000	0.4175	0.1308	0.0699	0.0327	0.0207	
	150	1.0000	0.4259	0.1363	0.0732	0.0349	0.0221	
	180	1.0000	0.4317	0.1405	0.0772	0.0378	0.0238	
	210	1.0000	0.4360	0.1097	0.0800	0.0383	0.0259	
	30	1.0000	0.6913	0.1863	0.0812	0.0385	0.0217	
	60	1.0000	0.6938	0.2249	0.0999	0.0483	0.0293	
	90	1.0000	0.5956	0.2309	0.1064	0.0501	0.0320	
$D^{5)}$	120	1.0000	0.5828	0.2306	0.1113	0.0538	0.0343	
	150	1.0000	0.5519	0.2183	0.1085	0.0540	0.0342	
	180	1.0000	0.5561	0.2409	0.1203	0.0588	0.0369	
	210	1.0000	0.5317	0.2448	0.1205	0.0603	0.0379	

<sup>&</sup>lt;sup>1)</sup>Control: unirradiated sample

in viscosity in black and white pepper was guessed similar to that of starch explained by other researchers <sup>20-26)</sup>

# Parameter values derived from viscosity, and the regression expressions and coefficients of irradiated black and white pepper.

To remove the affected variation for viscosity, the parameter values derived from the viscosity of black and white pepper are listed in Table 5. Hayashi et al.<sup>12)</sup> reported that the viscosity divided by the starch content to calculate a normalized parameter provides a more consistent response to irradiation treatment than any other viscosity values. The parameter values of irradiated black

and white pepper showed a dose dependent relation between unirradiated and irradiated samples and indicated that all of the values for the unirradiated samples were higher than the irradiated ones. A normalized parameter of the samples is a better parameter for detecting irradiation treatment than the viscosity value itself because the moisture and starch contents of black and white pepper have an influence on viscosity. Therefore, we expect that the viscosity (parameters A, B, and C) divided by moisture, starch content, and the control of parameter B provide detection values that reduce the fluctuation of viscosity values among the pepper samples. The regression expressions and coefficients of irradiated

<sup>&</sup>lt;sup>2)</sup>A: Black pepper prepared to 10% concentration

<sup>&</sup>lt;sup>3)</sup>B: Black pepper prepared to 13% concentration

<sup>&</sup>lt;sup>4)</sup>C: White pepper prepared to 7% concentration

<sup>&</sup>lt;sup>5)</sup>D: White pepper prepared to 10% concentration

Table 6. Regression expressions and coefficients of black pepper and white pepper prepared with different concentrations at various rpms

i pinis				
rpm	A <sup>1)</sup>	$B^{2)}$	$C^{3)}$	$\overline{\mathbf{D}}^{4)}$
30	$y = -363.21x + 4471.0$ $R^2 = 0.9688$	$y = -845.85x + 9774.0$ $R^2 = 0.9673$	$y = 4611.0e^{0.4149x}$ $R^2 = 0.9825$	$y = 10816e^{-0.3239x}$ $R^2 = 0.9660$
60	$y = -223.46x + 2836.0$ $R^2 = 0.9660$	$y = -533.63x + 6201.0$ $R^2 = 0.9675$	$y = 3199.0e^{-0.3868x}$ $R^2 = 0.9708$	$y = 7003.0e^{-0.2983x}$ $R^2 = 0.9708$
90	$y = -164.02x + 2145.0$ $R^2 = 0.9562$	$y = -428.33x + 4911.0$ $R^2 = 0.9770$	$y = 2577.0e^{-0.3689x}$ $R^2 = 0.9695$	$y = 5985.0e^{-0.2953x}$ $R^2 = 0.9695$
120	$y = -131.29x + 1769.0$ $R^2 = 0.9531$	$y = -351.33x + 4036.0$ $R^2 = 0.9731$	$y = 2208.0e^{-0.3546x}$ $R^2 = 0.9714$	$y = 5116.0e^{-0.2887x}$ $R^2 = 0.9932$
150	$y = -110.22x + 1523.0$ $R^2 = 0.9427$	$y = -299.90x + 3445.0$ $R^2 = 0.9707$	$y = 1972.0e^{-0.3480x}$ $R^2 = 0.9890$	$y = 4874.0e^{-0.2929x}$ $R^2 = 0.9959$
180	$y = -96.133x + 1350.0$ $R^2 = 0.9385$	$y = -265.69x + 3070.0$ $R^2 = 0.9727$	$y = 1793.0e^{-0.3407x}$ $R^2 = 0.9877$	$y = 2614.8e^{-0.2816x}$ $R^2 = 0.9972$
210	$y = -86.771x + 1223.0$ $R^2 = 0.9369$	$y = -239.53x + 2753.0$ $R^2 = 0.9666$	$y = 1653.0e^{-0.3380x}$ $R^2 = 0.9878$	$y = 2497.5e^{-0.2804x}$ $R^2 = 0.9990$

<sup>&</sup>lt;sup>1)</sup>A: Black pepper prepared to 10% concentration <sup>2)</sup>B: Black pepper prepared to 13% concentration

black and white pepper are listed in Table 9. The regression coefficients of irradiated black and white pepper showed very high relationship between irradiation dose and viscosity. This trend was similar for all stirring speeds.

#### **CONCLUSION**

All samples indicated a decrease of viscosity and standard deviation by increasing the stirring speeds. Viscosity is dependent on the concentration of the suspended white and black pepper and detection was possible for all concentrations. These results suggest that the detection of irradiated black and white pepper at various stirring speeds and different concentrations is possible by the viscometric method.

#### **ACKNOWLEDGEMENTS**

The authors would like to thank the Ministry of Science and Technology (Research of the Long-and-Midterm Nuclear R & D Program) for financial support during this study.

#### 국문요약

본 연구는 점도 측정법을 이용하여 방사선 조사된 검은 후추가루와 흰 후추가루의 조사유무를 확인하기 위해 이전의 연구에서 수행되지 않은 다양한 rpm에서의 검지 가능성과 회귀식 및 회귀계수를 설정하여 보다 최적화 되고 체계적인 검지 기술을 확립하기 위해 수행되어졌다. 실험에 사용된 검은 후추가루와 흰 후추가루는 polyethylene bag에 포장되어졌고 한국원자력연구소내의 Co-60 감마선 조사시설을 이용하여 2.5, 5, 7.5, 10, 15 kGy로 조사되어졌다. 10 %, 13% 농도의 검은 후추가루와 7%, 10% 농도의 흰 후추가루 시료를 만들기 위하여 증류수를 가하여 현탁시킨 후에 알카리화 하였고 Brookfield DV-III rotation viscometer 를 이용하여 30℃에서 30초 동안 30, 60, 90, 120, 150, 180, 210 rpm 에서 점도를 측정하였다. 모든 시료의 점도와 표준편차는 조사선량과 rpm이 증가할수록 감소하는 경향을 보였으며, 농도가 증가할수록 점도도 증가되어 농도 의존적인 경향을 보여 주었고, 이러한 경향은 모든 시료에서 유사하게 나타났다. 120 rpm 에서 10% black pepper, 13% black pepper, 7% white pepper, 10% white pepper 의 회귀식과 회귀계수는 0.9531 (y=−131.29x+1769.0), 0.9725 (y=−351.33x+4036.0), 0.9731 (y=208.0e<sup>-0.3846x</sup>), 및 0.9959 (y=5116.0e<sup>-0.2887x</sup>)를 나타내었으며 조사선량과 점도간의 높은 상관성을 보여 주었다. 이상

<sup>&</sup>lt;sup>3</sup>C: white pepper prepared to 7% concentration <sup>4</sup>D: white pepper prepared to 10% concentration

y: viscosity x: irradiation dose

의 결과를 종합하여 볼 때 점도 측정법은 방사선 조사된 검은 후추가루와 흰 후추가루의 조사유무를 확인할 수 있는 유용한 방법임을 확인할 수 있었다.

#### **REFERENCES**

- Schreiber, G. A., Leffke, A., Mager, M., Helle, N. and B gl, K. W.: 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 (1994)
- 2. Hayashi, T., Todoriki, S. and Kohyama, K.: Irradiation effects on pepper starch viscosity. *J. Food Sci.*, **59**, 118-120 (1994)
- de Alwis, H. M. G. and Grandison, A. S.: Viscometry as a detection method for electron beam irradiation of black pepper. *Food Control*, 3, 205-208 (1992)
- Barabasy, S., Sharif, M., Farkas, J., Felf ldi, J., Koncz., Formanek, Z. and kaffka, K.: Attempts to elaborate detection methods for some irradiated food an dry ingredients. In *Detection Methods for Irradiated Foods* (McMurray, C. H., Stewart, E. M., Gray, R. and Pearce, J. ed), The Royal Society of Chemistry, Cambridge, p.185-201 (1996)
- Diehl, J. F.: Safety of irradiated foods. Marcel Dekker, Inc., New York. p. 339-352 (1995)
- Hayashi, T. and Todoriki, S.: Detection of irradiated peppers by viscosity measurement at extremely high pH. Radiat. Phys. Chem., 48, 101-104 (1996)
- Farkas, J., Sharif, M. M. and Koncz, A.: Detection of some irradiated spices on the basis of radiation induced damage of starch. Radiat. Phys. Chem., 36, 621-627 (1990)
- Yang, J. S.: General survey of detection methods for irradiation foods. J. Korean Nuclear Society, 29, 500-507 (1997)
- Rahman, R., Haque, A. K. M. M. and Sumar, S. Chemical and biological methods for the identification of irradiated foodstuffs. Nutr. & Food Sci. 2, 4-11 (1995)
- Heide, L. and B gl, K. W.: Detection methods for irradiated ood-luminescence and viscosity measurements. Int. J. Radiat. Biol., 57, 201-219 (1990)
- B gl, K. W.: Identification of irradiated foods-methods, development and concepts. *Appl. Radiat. Isot.*, 40, 1203-1210 (1989)
- Farkas, J., Koncz, A. and Sharif, M. M.: Identification of irradiated dry ingredients on the basis of starch damage. *Radiat. Phys. Chem.*, 35, 324-328 (1990)

- Hayashi, T., Todoriki, S. and Kohyama, K.: Applicability of viscosity measuring method to the detection of irradiated spices. *Nippon Shokuhin Kogyo Gakkaishi*, 40, 456-460 (1993)
- 14. Hayashi, T., Todoriki, S. and Kohyama, K.: Applicability of viscosity measurement to the detection of irradiated peppers. In Detection Methods for Irradiated Foods (McMurray, C. H., Stewart, E. M., Gray, R. and Pearce, J. ed), The Royal Society of Chemistry, Cambridge, p.215-225 (1996)
- Hayashi, T.: Collaborative study of viscosity measurement of black and white peppers. In *Detection Methods for Irradiated Foods* (McMurray, C. H., Stewart, E. M., Gray, R. and Pearce, J. ed), The Royal Society of Chemistry, Cambridge, p.229-237 (1996)
- 16. Heide, L., N rnberger, E. and B gl, K. W.: Investigations on the detection of irradiated food measuring the viscosity of suspended spices and dried vegetables. *Radiat. Phys. Chem.*, 36, 613-619 (1990)
- Sharif, M. M. and Farkas, J.: Analytical studies into radiation-induced starch damage in black and white pepers. *Radiat. Phys. Chem.*, 42, 383-386 (1993)
- Köksel, H., Celik, S. and Tuncer, T.: Effects of gamma irradiation on durum wheats and spaghetti quality. Cereal Chem., 73, 506-509 (1996)
- Stephen A. M.: Food polysaccharides and their applications. Marcel Dekker, Inc., New York. p. 67-97 (1995)
- Komiya, T., Yamada, T., Kawakishi, S. and Nara, S.: Effect of linseed oil on the physico-chemical properties of potato and corn starches during gamma-irradiation. J. Jap. Soc. Starch Sci., 29, 1-6 (1982)
- Bhatty, R. S. and Macgregor, A. W.: Gamma irradiation of hulless barley: effect on grain composition, β- glucans and starch. Cereal Chem., 68, 463-469 (1988)
- Kang, I. J., Byun, M. W., Yook, H. S., Bae, C. H., Lee, H. S., Kwon, J. H. and Chung, C. K.: Production of modified starches by gamma irradiation, Radiat. *Phys. Chem.*, 54, 425-430 (1999)
- MacArthur, L. A. and D'Appolonia, B. L.: Gamma radiation of wheat. II. Effects of low-dosage radiations on starch properties, *Cereal Chem.*, 61, 321-326 (1984)
- Roushdi, M., Harras, A., El-meligi, A. and Bassim,
   M.: Effect of high doses of gamma rays on corn

- grains. Staerke 35, 15-21 (1983)
- 25. Sabularse, V. C., Liuzzo, J. A., Rao, R. M. and Grodner, R. M.: Physicochemical characteristics of brown rice as influenced by gamma irradiation. *J. Food Sci.*, 57, 143-145 (1992)
- 26. Sokhey, A. S. and Hanna, M. A.: Properties of irradiated starches. *Food Structure* 12, 397-410 (1993)
- A.O.A.C. Official Methods of Analysis, 16th ed.: Association of Official Analytical Chemists, Washington, DC., p.31 (1995)
- 28. Hayashi, T. and Kawashima, K.: The effect of gamma irradiation on the sucrose content in sweet potato roots and potato tubers. *Agric. Biol. Chem.*, **46**, 1475-1479 (1982)