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DETECTION OF SOME IRRADIATED FOODS BY THERMOLUMINESCENCE AND VISCOSITY METHODS

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ABSTRACT: **The present investigation was carried out to establish a detection method for irradiated black pepper and marjoram through thermoluminescence (TL) and wheat, cinnamon and ginger through viscosity measurement. All samples were packed in polyethylene bags then irradiated at 5, 10 and 15 kGy for black pepper, marjoram, cinnamon and ginger. Wheat was irradiated at 1, 2 and 3 kGy. All samples were stored for eight months at room temperature. Results indicated that irradiation treatment caused markedly increasing in TL intensity for irradiated black pepper and marjoram while irradiation treatment decreased apparent viscosity of wheat flour, cinnamon and ginger powder, post irradiation and during storage. Therefore it could be concluded that the TL analysis can be used to detect irradiated black pepper and marjoram, also the viscosity measurement can be used to detect irradiate wheat flour, cinnamon and ginger powder than unirradiated ones after irradiation process and also during 8 months of storage at ambient temperature.**

Key words: Thermoluminescence, viscosity, identification, wheat, ginger, cinnamon

INTRODUCTION

The ionizing radiation treatment is a safe and reliable method widely accepted and used for food preservation (WHO, 1981, WHO, 1994, and WHO, 1999). Food irradiation is a processing technique used to eliminate the presence of contaminating microorganisms, as an alternative to chemical products that may result dangerous for human and have a negative impact on the environment (IFST, 2006). In particular, dried herbs and spices are among the most commonly irradiated food products to prevent losses during storage due to infestation and putrefaction (Calderón *et al.*, 1995). Maximum absorbed doses of 10 kGy have been recommended (IFST, 2006 and IAEA/FAO, 2006) but not all the countries apply the same legislation concerning procedures and labeling requirements. Therefore, reproducible, reliable and simplest as possible methods to identify irradiated samples and estimate the received dose in the corresponding range are essential to control whether or not the appropriate regulations are being satisfied. The commercialization of irradiated food strongly depends on the availability of sensitive and reliable analytical techniques to identify irradiated food stuffs. Among the physical identification methods, the thermoluminescence (TL), is suitable for food containing silicate minerals as contaminants (Bayram and Delincée, 2004), such as herbs (Calderón *et al.*, 1995) and spices (Sharifzadeh and Sohrabpour, 1993). Viscosity studies were performed with 20 different spices or dried vegetables (Heide *et al.*, 1988). With cinnamon, ginger, black and white pepper the low viscosity values permit the conclusion that the samples have most probably been irradiated and the high viscosity values indicate unirradiated samples. Further lots were investigated to estimate the variations of viscosity, depending on the origin of the samples. Additional storage experiments showed that measuring the viscosity may be a simple method to identify some radiation treated spices even after years, but performance of viscosity studies on spices are so far more or less empirical. No general concept for preparing the suspensions of individual spice types can be given. The preliminary examinations require further refinement and standardization (Bögl, 1990). The viscosity of spices may be influenced mainly by the pH-value, temperature and concentration of the suspension, the soaking time and grain size. Therefore these parameters require careful consideration when preparing the spice suspensions

(Heide *et al.*, 1988; Mohr *et al.*, 1988).Therefore the aim of the present work was to study the applicability of the TL analysis and viscosity measurement as methods for detecting the irradiated black pepper and marjoram, and wheat, cinnamon and ginger, respectively post irradiation treatment and during storage at ambient temperature.

MATERIALS AND METHODS

Materials

Black pepper, marjoram, cinnamon, ginger and wheat were purchased from a local market, Zagazig City, Egypt. Samples were packed in polyethylene bags, each bag contains 150 g for black pepper or marjoram and 100 g for wheat, cinnamon or ginger.

Irradiation Treatments

All samples were irradiated using a ^{60}Co Russian gamma chamber, (dose rate 3.7 kGyhr $^{-1}$), at the Nuclear Research Center, Atomic Energy Authority, Abu Zaabal, Egypt. The applied radiation doses were 5, 10, and 15 kGy, for black pepper, marjoram, cinnamon and ginger and 1, 2 and 3 kGy for wheat. The samples were divided into two groups, the first one was black pepper and marjoram for the TL analysis however viscosity measurement applied on the second part which contain wheat, cinnamon and ginger, then stored for eight months at room temperature for measuring periodically every two month.

Methods

Thermoluminescence (TL)

Preparation of minerals extract

The established method for separating a mineral extract from spice sample was used according to the procedure recommended by the European Committee for Standardization (EN 1788, 1997). The following main steps were followed:

The 150 g samples were agitated with 2 L distilled water for 5 min, and then sieved through a 250 µm nylon cloth and the constituents retained were discarded. The filtered solution was allowed to settle for about 5 min in order to separate the sediment minerals from 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 gmL^{-1} by an addition of deionized 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 off. This procedure was repeated until all the organic materials were removed. After the polytungstate solution was removed, the minerals were washed twice with water and pelleted through centrifugation at 1000 rpm, and acidified 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 with 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 2 mL of acetone twice and dried in a laboratory oven at 50°C for 3 h.

The dried minerals (1mg) 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 (EN 1788, 1997).

Thermoluminescence (TL) measurement

TL measurement was carried out using a TL reader (Harshaw 4000, Germany) with heat ranging from 50 to 350°C at a heating rate of 6 °C s^{-1} and hold at 350°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 analysis (Glow 1), the discs were irradiated with a dose of 5 kGy and the second glow analysis (Glow 2) under taken after irradiation.

Calculation

The TL ratio (Glow1 /Glow 2) was then determined.

Viscosity Measurement

Prior to viscosity measurement, the samples were ground to size smaller than 0.5 nm. The viscosity was measured with a Brookfield DV – I viscometer Germany) according to Hayashi *et al.* (1993, 1995) with a slight modification according to Heide *et al.* (1990), as follows:

Suspensions of cinnamon; ginger and wheat (20, 10 and 8 % w/v, respectively) were homogenized with a Universal laboratory aid type 309 (Mechanika precyzyjna Japan) for 30 s at the 12000 rpm, the pH was adjusted to 13.0 with 33% NaOH and the suspensions were heated in boiling water with an occasional agitation for 30 min, followed by incubation for 3 h at

25°C. The viscosity of the suspension was measured at 25°C and 30 rpm with the Brookfield DV – I viscometer, the apparent viscosity ($mPas^{-1}$) was measured after 30s.

RESULTS AND DISCUSSION

Thermoluminescence (TL)

The general patterns of irradiated black pepper and marjoram (first and second glow curves) are shown in Fig. 1 and 2, respectively. It is clear that the first glow curve was not found for unirradiated black pepper or marjoram while it is clearly appear for irradiated samples, owing to the TL intensities of minerals separated from irradiated samples were higher than those of unirradiated samples. These results agreed with those obtained by (Khan *et al.*, 1998). On the other hand, the second glow curve of all samples under investigation was showed regions higher than those of the first glow curve, this due to TL emission occurs when the excited electrons (by irradiation) return to the original level at a certain temperature (Schreiber *et al.*, 1995). It is obvious that the TL intensities of irradiated marjoram were higher than those of irradiated black pepper at the same doses. These results agreed with those obtained by (Sang-Duk *et al.*, 2000), who observed the same phenomena among the species and these differences seemed to have been caused by the differences in mineral composition and size used for TL measurement. In addition, the glow curve ratios as well as an analysis of glow curve shapes were preferred for identifying irradiated and unirradiated samples in several studies (Khian and Delincée, 1995). Table 1 and Fig. 3 and 4 demonstrate the glow curve ratios for unirradiated and irradiated black pepper and marjoram, respectively. It is clearly that the glow curve ratios of the unirradiated black pepper and marjoram were less than 0.1 (varied between 0.0016 and 0.0035), so they were classified as unirradiated samples. Since the glow curve ratios for irradiated black pepper and marjoram were greater than 0.50 (varied between 0.53 and 1.14) and the typical glow shapes for irradiated samples were observed, therefore these samples were classified as irradiated samples. It could be concluded that TL analysis can be used to detect irradiated black pepper and marjoram than unirradiated ones after irradiation process and also during 8 months of storage at ambient temperature. Moreover, the study indicate that samples stored for 8 months after irradiation could still be sufficiently distinguished from non- irradiated samples.

Viscosity

Wheat flour

Data in Table 2a and Fig. 5 show that the apparent viscosity value of unirradiated wheat flour (control) was 455 mPa s^{-1} at 30 rpm and 25°C. It is obvious that gamma irradiation doses caused a markedly decrease in apparent viscosity values of treated samples, as a function of radiation dose, where reached to 345.4, 171.5 and 153 mPa s^{-1} corresponding 24%, 62% and 66 % decrease percentages for irradiated wheat flour at doses 1, 2 and 3 kGy, respectively. These results agreed with those obtained by (Hayashi and Todoriki, 1996) who applied viscosity measurement on black pepper and observed same influence for gamma irradiation. The reduction of viscosity in irradiated wheat flour due to the effect of the free radicals created by gamma irradiation on starch molecule, free radicals are responsible for starch molecular changes such as the uncoiling of starch chain and fragmentation by breaking of the hydrogen bonds in the starch molecules. These changes may affect the physical and rheological properties, i.e. decreasing viscosity of foods containing high amount starchsuch as cereals (MacArthur and D'Appolonia, 1984; Sokhey and Hanna, 1993). During storage at ambient temperature, the apparent viscosity values of unirradiated wheat flour were decreased during storage for 8 month, by 4.1% after 8 months. However, the decreasing percentages of apparent viscosity for irradiated samples were higher than those of unirradiated samples where reached to 8.1 %, 12.4 % and 20.8 % after 8 months of storage for irradiated samples at dose levels of 1, 2 and 3 kGy, respectively (Table 2b and Fig. 5). Depending on the above mentioned results, it could be concluded that the viscosity measurement can be used to detect irradiated wheat flour than unirradiated ones relatively after irradiation process and also can be used as a method for detecting irradiated wheat flour during 8 months of storage.

Cinnamon powder

The apparent viscosity values of unirradiated cinnamon were significantly higher than those of irradiated when the viscosity determined at 30 rpm, 25 °C and incubation for 3 h at 25 °C (Table 3a and Fig. 6) where the apparent viscosity value of unirradiated sample was 370 mPa s^{-1} while the apparent viscosity values of irradiated samples at doses 5, 10 and 15 kGy were 190.2, 140.4 and110.2 mPa s^{-1}, respectively.These results showed that the irradiation treatment have been clearly reduced the apparent viscosity of cinnamon by 48%, 62% and 70% at doses 5, 10 and 15 kGy, respectively. These results agreed with those obtained by (Heide *et al.*, 1990), who reported that the viscosity values of irradiated cinnamon powder decreased by gamma irradiation treatment. The reduction in apparent viscosity in samples under investigation may be due to the changes in shape, molecular weight and structure of major compounds (macromolecules) such as, starch, pectin and cellulose of foods where the structure of which will be injured by ionizing radiation. Consequently, alterations of the gelatinization properties occur (Samec, 1958). Effect of storage periods on apparent viscosity of unirradiated cinnamon powder showed in Table 3b and Fig. 6, it is clear that the apparent viscosity of unirradiated samples were decreased during storage periods by 1.5%, 3.9%, 7.1% and 9.1% after 2, 4, 6 and 8 months, respectively. However, the decreasing in apparent viscosity values of irradiated samples were higher than those unirradiated samples where reduced by 13.8%, 22% and 32.8% after 8 months of storage at doses 5, 10 and 15 kGy, respectively. From these results it can be said that the viscosity measurement is the appropriate technique for detecting cinnamon irradiated or not even 8 months of storage.

Ginger powder

Effect of gamma irradiation and storage periods on apparent viscosity of ginger powder were showed in Tables 4a and 4b and Fig. 7. Tested irradiated ginger samples showed decreased apparent viscosity value as compared with the corresponding unirradiated sample, where was 620.5 mPa s^{-1} for unirradiated and 235.8, 101 and 44.9 mPa s^{-1} for irradiated ones at doses 5, 10 and 15 kGy, respectively. Viscosity was reduced as dose levels were increased, where the decrease percentages reached to 61%, 82% and 92% with dose levels 5, 10, and 15 kGy, respectively. The same results obtained by (Heide *et al.*,1990). During storage the apparent viscosity values wer reduced by .5%, 1.8%, 3.6% and 4.6% after 2, 4, 6 and 8 months, respectively for untreated samples. However these percentages of irradiated samples at doses 5, 10, and 15 kGy were higher where reached to 15.6%, 37.6% and 60.5% in the end of storage at ambient temperature. Viscosity measurements of ginger powder may be an indicator for radiation exposure even 8 months of storage.

Table 1. TL ratio (Glowl/Glow2) of unirradiated and irradiated black pepper and marjoram during storage

Storage period (month)	Spices	Irradiation dose (kGy)			
		0	5	10	15
0	Black pepper	0.0035	0.56	0.61	0.77
2		0.0034	0.57	0.66	1.14
4		0.0034	0.63	0.71	0.60
6		0.0030	0.53	0.60	0.64
8		0.0025	0.58	0.63	0.67
0	Marjor am	0.0028	0.68	0.71	0.69
2		0.0019	0.77	0.74	0.65
4		0.0016	0.68	0.64	0.62
6		0.0026	0.65	1.01	0.51
8		0.0022	0.72	0.77	0.75

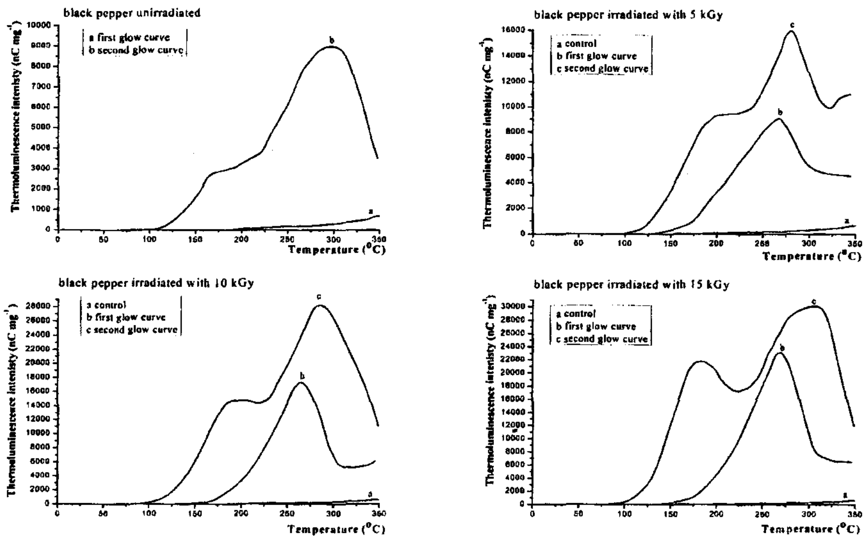

Fig. 1. Thermoluminescence intensity of mineral extracted from black pepper either unirradiated or irradiated with 5, 10 and 15 kGy at zero time

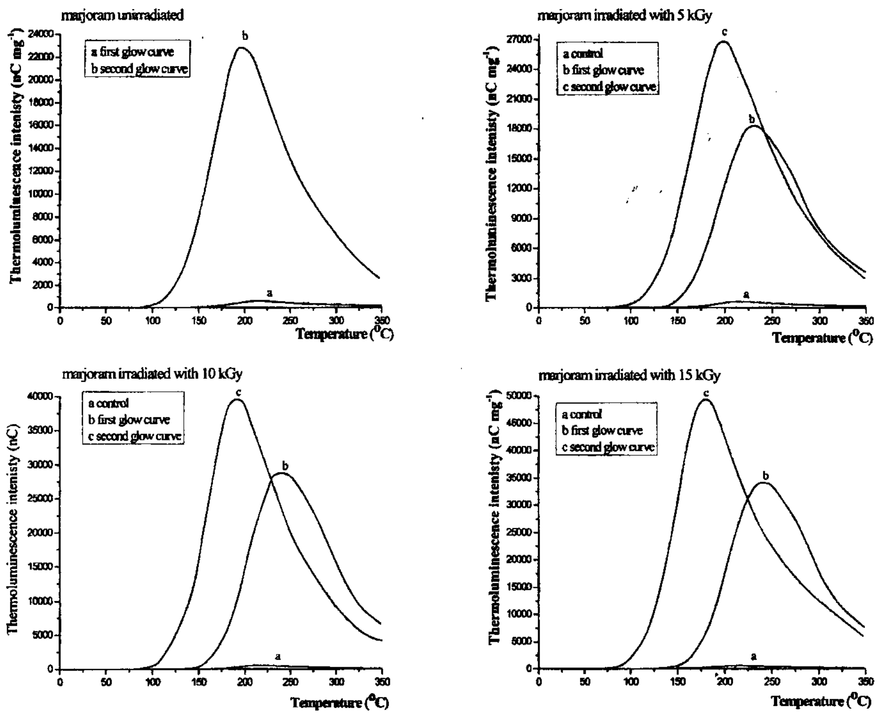

Fig. 2. Thermoluminescence intensity of mineral extracted from marjoram either unirradiated or irradiated with 5, 10 and 15 kGy at zero time

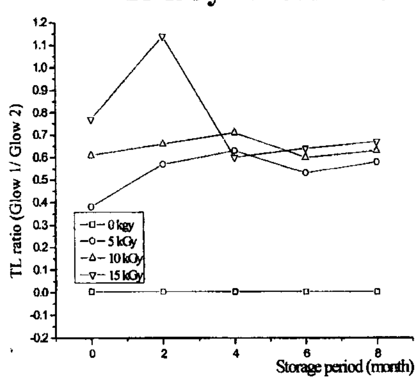

Fig. 3. TL ratio (Glow1/Glow2) of unirradiated and irradiated black pepper

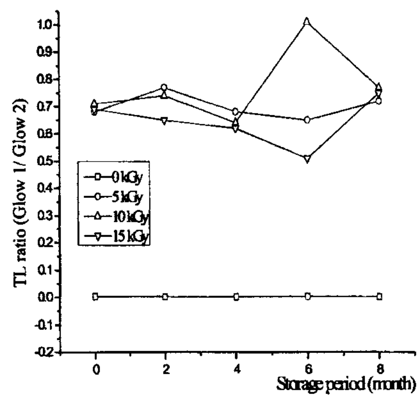

Fig. 4. TL ratio (Glow1/Glow2) of unirradiated and irradiated marjoram

Table 2a. Effect of gamma irradiation on apparent viscosity (mPa s^{-1}) of gelatinized wheat flour during storage at ambient temperature

Storage period (month)	Irradiation dose (kGy)							
	0		1		2		3	
	Visc. mPa s^{-1}	D. *%	Visc. mPa s^{-1}	D. *%	Visc. mPa s^{-1}	D. *%	Visc. mPa s^{-1}	D. *%
0	455.0	-	345.4	24	171.5	62	153.0	66
2	450.0	-	338.3	24	162.5	63	142.0	68
4	441.0	-	335.0	24	162.5	63	133.0	69
6	439.5	-	325.6	25	154.4	64	126.0	71
8	436.2	-	317.3	27	150.2	65	121.1	72

* = Decrease %

Table 2b. Effect of storage period on apparent viscosity (mPa s^{-1}) of irradiated gelatinized wheat flour during storage at ambient temperature

Storage period (month)	Irradiation dose (kGy)							
	0		1		2		3	
	Visc. mPa s^{-1}	D. *%	Visc. mPa s^{-1}	D. *%	Visc. mPa s^{-1}	D. *%	Visc. mPa s^{-1}	D. *%
0	455.0	-	345.4	-	171.5	-	153.0	-
2	450.0	1.0	338.3	2.0	162.5	05.2	142.0	07.1
4	441.0	3.0	335.0	3.0	162.5	05.2	133.0	13.0
6	439.5	3.4	325.6	5.7	154.4	09.9	126.0	17.6
8	436.2	4.1	317.3	8.1	150.2	12.4	121.1	20.8

* = Decrease %

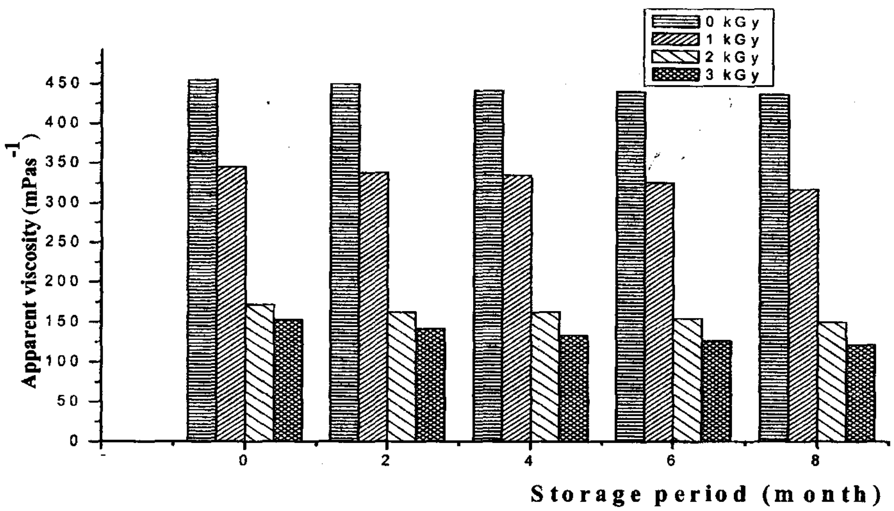

Fig. 5. Effect of gamma irradiation on apparent viscosity (mPa s^{-1}) of gelatinized wheat flour during storage at ambient temperature

Table 3a. Effect of gamma irradiation on apparent viscosity (mPa s^{-1}) of gelatinized cinnamon powder during storage at ambient temperature

Storage period (month)	Irradiation dose (kGy)							
	0		5		10		15	
	Visc. mPa s^{-1}	D. *%	Visc. mPa s^{-1}	D. *%	Visc. mPa s^{-1}	D. *%	Visc. mPa s^{-1}	D. *%
0	370.0	-	190.2	48	140.4	62	110.2	70
2	364.1	-	184.5	49	134.9	62	103.1	71
4	355.6	-	177.3	50	124.8	64	093.9	73
6	343.7	-	170.8	50	115.5	66	081.9	76
8	336.3	-	163.9	51	109.5	67	074.0	77

* = Decrease %

Table 3b. Effect of storage period on apparent viscosity (mPa s^{-1}) of irradiated gelatinized cinnamon powder during storage at ambient temperature

Storage period (month)	Irradiation dose (kGy)							
	0		5		10		15	
	Visc. mPa s^{-1}	D. *%	Visc. mPa s^{-1}	D. *%	Visc. mPa s^{-1}	D. *%	Visc. mPa s^{-1}	D. *%
0	370.0	-	190.2	-	140.4	-	110.2	-
2	364.1	1.5	184.5	03.0	134.9	03.9	103.1	06.4
4	355.6	3.9	177.3	06.7	124.8	11.1	93.9	14.7
6	343.7	7.1	170.8	10.1	115.5	17.7	81.9	25.6
8	336.3	9.1	163.9	13.8	109.5	22.0	74.0	32.8

* = Decrease %

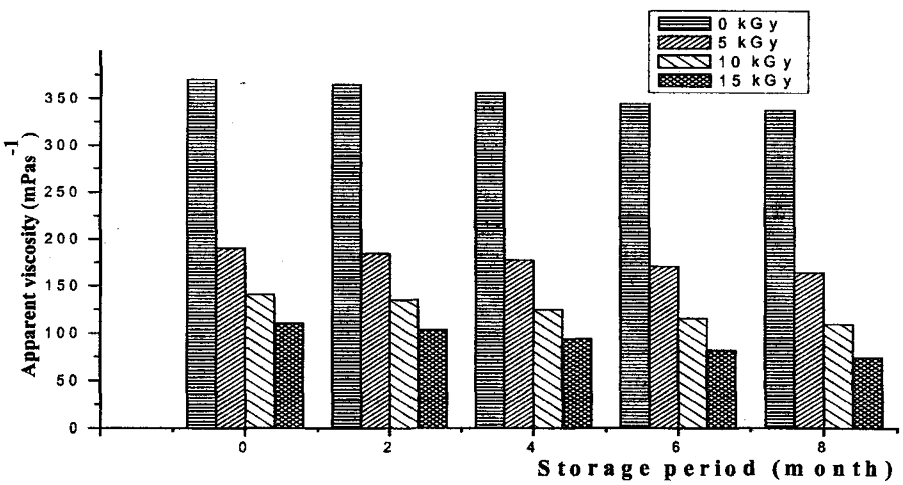

Fig. 6. Effect of gamma irradiation on apparent viscosity (mPa s^{-1}) of gelatinized cinnamon powder during storage at ambient temperature

Table 4a. Effect of gamma irradiation on apparent viscosity (mPa s^{-1}) of gelatinized ginger powder during storage at ambient temperature

Storage period (month)	Irradiation dose (kGy)							
	0		5		10		15	
	Visc. mPa s^{-1}	D. *%	Visc. mPa s^{-1}	D. *%	Visc. mPa s^{-1}	D. *%	Visc. mPa s^{-1}	D. *%
0	620.5	-	235.8	61	101.0	82	44.9	92
2	616.9	-	228.8	62	94.3	84	39.9	93
4	609.1	-	220.8	63	82.3	86	33.9	94
6	597.6	-	205.0	65	70.8	88	20.7	96
8	591.7	-	198.8	66	63.0	89	17.7	97

* = Decrease %

Table 4b. Effect of storage period on apparent viscosity (mPa s^{-1}) of irradiated gelatinized ginger powder during storage at ambient temperature

Storage period (month)	Irradiation dose (kGy)							
	0		5		10		15	
	Visc. mPa s^{-1}	D. *%	Visc. mPa s^{-1}	D. *%	Visc. mPa s^{-1}	D. *%	Visc. mPa s^{-1}	D. *%
0	620.5	-	235.8	-	101.0	-	44.9	-
2	616.9	0.5	228.8	02.9	94.3	06.6	39.9	11.1
4	609.1	1.8	220.8	06.3	82.3	18.5	33.9	24.4
6	597.6	3.6	205.0	13.0	70.8	29.9	20.7	53.8
8	591.7	4.6	198.8	15.6	63.0	37.6	17.7	60.5

* = Decrease %

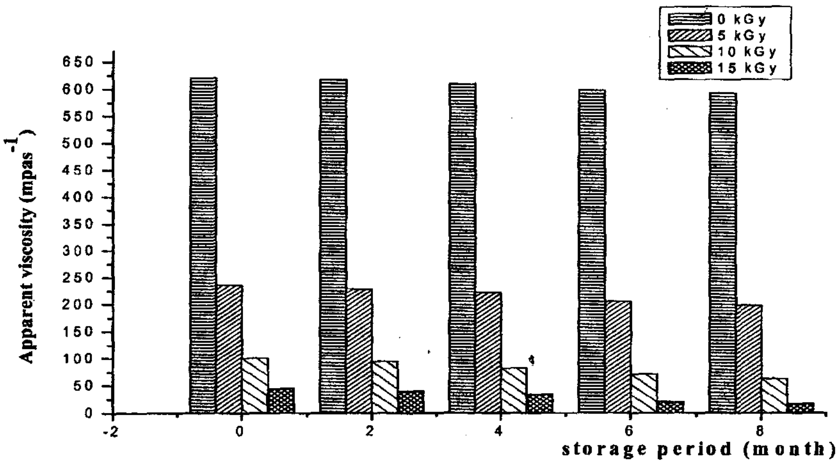

Fig. 7. Effect of gamma irradiation on apparent viscosity (mPa s^{-1}) of gelatinized ginger powder during storage at ambient temperature

Conclusion

From our investigation on the identification of irradiated foods with TL and viscosity analysis, it could be concluded that irradiation treatment caused markedly increase in TL intensity for irradiated black pepper and marjoram while caused decrease in apparent viscosity for irradiated wheat flour, cinnamon powder and ginger powder after irradiation process and during storage. Therefore the identification of irradiated black pepper and marjoram could be determined on the basis of the TL response of the mineral extract and the values of glow curve ratio also could be very important in the detection of irradiated spices in our study. Also the viscosity measurement can be used to detect irradiated wheat flour, cinnamon powder and ginger powder after irradiation process and during storage at ambient temperature. Therefore, both TL and viscosity methods are extremely powerful technique for identification such irradiated dry foods.

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التعرف على بعض الأغذية المعاملة بالإشعاع بطريقتي الوميض الحراري و اللزوجة

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١- قسم علوم الأغذية- كلية الزراعة- جامعة الزقازيق

٢- هيئة الطاقة الذرية- مركز البحوث النووية- أبو زعبل- ص.ب ١٣٧٥٩ مصر

أجريت هذه الدراسة للتعرف على المعاملة الإشعاعية للفلفل الأسود و البردقوش من خلال طريقة الوميض الحراري، وطحين القمح، القرفة والزنجبيل من خلال مقياس اللزوجة. حيث تم تعبئة جميع العينات في أكياس من البولي إيثيلين وتم تشعيع كل من الفلفل الأسود والبردقوش وطحين القرفة والزنجبيل بجرعات ٥، ١٠و ١٥ كيلو جراى و طحين القمح بجرعات١، ٢ و٣ كيلو جراى. ثم خزنت جميع العينات على درجة حرارة الغرفة لمدة ٨ أشهر.

وقد أظهرت النتائج المتحصل عليها أن المعاملة الإشعاعية قد أدت إلى زيادة ملحوظة فى قراءات الوميض الحراري لعينات الفلفل الأسود والبردقوش بينما انخفضت اللزوجة الظاهرية لعينات طحين القمح و القرفة والزنجبيل انخفاضا ملحوظا عقب المعاملة الإشعاعية و خلال فترة التخزين. لذا يمكن استنتاج أنه يمكن استخدام كل من طريقة الوميض الحراري للتعرف على الفلفل الأسود و البردقوش المعامل بالإشعاع وطريقة اللزوجة للتعرف على مطحون القمح و القرفة والزنجبيل المعامل بالإشعاع عقب المعاملة الإشعاعية و خلال ٨ أشهر من التخزين على درجة حرارة الغرفة.