Gamma radiation effects in coriander (coriandrum sativum L) for consumption in Mexico

E. Cruz-Zaragoza^a, B. Ruiz-Gurrola^b, C. Wacher^c, T. Flores Espinosa^c, M. Barboza-Flores^d ^aUnidad de Irradiación y Seguridad Radiológica, Instituto de Ciencias Nucleares, Universidad Nacional Autónoma de México, Apartado Postal 70-543, México, D.F., 04510, México. ^bDepartamento de Agricultura y Ganadería, Apartado Postal 305, Universidad de Sonora, Hermosillo Sonora, 83190, México. ^cDepartamento de Alimentos y Biotecnología, Facultad de Química, Universidad Nacional Autónoma de México, México, D.F., 04510, México. ^dCentro de Investigación en Física, Universidad de Sonora, Apartado Postal 5-088, Hermosillo, Sonora, 83190, México.

Recibido el 10 de marzo de 2010; aceptado el 31 de agosto de 2010

Ionizing radiation is an effective process for disinfecting and prolonging the shelf-life of several food products. Food irradiation may be one of the most significant contributions to public health in the developing countries. Following the irradiation it is necessary to analyze the radiation dose effects in foodstuffs. Thermally stimulated luminescence (TL) properties and microbiological load as a function of the gamma doses were analyzed in fresh commercial Coriander (*Coriandrum sativum L*.) samples. For TL analyses the polymineral fraction was separated from coriander and 10 μ m size particles were selected. The polymineral samples were exposed to a 0.5-15,000 Gy dose from gamma radiation using a ⁶⁰Co facility, Gammabeam 651PT, semi-industrial irradiator with 98.4 Gy/min dose rate. The glow curves were broad bands and characteristic of quartz that is present in the sample as detected by XRD. The main TL characteristics were determined, including the structure of the glow curves, TL response, reproducibility of TL signals over 12 cycles of subsequent irradiations, and the fading effect during the storage during 30 days. The TL method was found useful for detection of irradiated coriander. In order to analyze the effect of gamma radiation on the bacterial load in the fresh food coriander, several coriander samples were exposed to 0-10 kGy dose. It was observed that at 0.5 kGy dose the aerobic mesophilic count was reduced to 99.9 %, while the initial total coliform bacteria decreased from 871,000 cfu/g to less than 100. The microbiological results are lower than the limit indicated by the Mexican regulatory authority; 150,000 cfu/g for mesophiles and 100 cfu/g for total coliforms. The aim of this work is to investigate the TL properties of the polymineral fraction obtained from coriander and to measure the microbiological load as a function of the gamma irradiation dose also.

Keywords: Food irradiation; coriander; polymineral fraction; radiation effects.

El proceso mediante radiación ionizante es muy efectivo para desinfestar y prolongar la vida de anaquel de varios productos alimenticios. La irradiación de alimentos puede contribuir significativamente a la salud pública en los países en desarrollo. Después de la irradiación, es necesario analizar los efectos de la dosis en los alimentos. Se analizan la propiedad de luminiscencia térmicamente estimulada y la carga microbiana en función de la dosis gamma del cilantro (*Coriandrum sativum L*.) fresco de muestras comerciales. Para el análisis TL, se separó la fracción polimineral del cilantro y se eligieron tamaños de partícula de 10 μ m. Las muestras poliminerales fueron expuestas a dosis entre 0.5-15,000 Gy de radiación gamma. Se utilizó el irradiador semi-industrial de ⁶⁰Co, Gammabeam 651PT, con razón de dosis de 98.4 Gy/min. Las curvas de brillo fueron bandas anchas y característica del cuarzo, que está presente en las muestras detectado por DRX. Se determinaron las características TL importantes, incluyendo la estructura de las curvas de brillo, la respuesta TL, reproducibilidad de las señales TL en un ciclo de irradiaciones sucesivas, y el efecto del fading durante el almacenamiento hasta 30 días. El método TL fue útil para la detección del cilantro irradiado. También con el fin de analizar el efecto de la radiación gamma en la carga bacteriológica del alimento fresco, las muestras los coliformes se redujeron de 871,000 ufc/g a menos de 100. Los resultados de microbiología encontrados al final de la dosis, están de acuerdo con los límites indicados para el consumo por la autoridad sanitaria en México; 150,000 ufc/g para mesófilos y 100 ufc/g en el caso de coliformes totales. El objetivo de este trabajo, es investigar las propiedades TL de la fracción polimineral obtenida del cilantro y también determinar de la radiación en la carga microbiana en función de la dosis de radiación gamma.

Descriptores: Irradiación de alimentos; cilantro; fracción polimineral; efectos de radiación

PACS: 61.80.-x; 61.80.Ed; 87.53.-j; 78.60.Kn

1. Introduction

The ionizing radiation processing for foodstuffs treatment is being adopted in many countries, mainly in the European Community [1-4] to eliminate insect infestation, microbial load and the reduction of pathogenic microorganisms instead of using conventional methods, such as fumigation with ethylene oxide, methyl bromide, drying, heating, etc. It is well documented that the fresh and dehydrated vegetables and spices have an enormous microbial load and different types would survive to the conventional methods applied [5-7]. In order to increase the level of safety in foods

for consumers, the number of countries that accepted irradiation of foods is increasing. The irradiation technology is more adequate since it is safer, economical and is not associated to toxicity in foods [3,8-10]. According to the Montreal Protocol, in the year 2015 developing countries must ban the fumigation method based in toxic gases for foods treatment [11,12] because of the risk to depleting the ozone layer and being carcinogenic to humans. However, in developing countries there is a lack investigation related to detection methods for specific foods treated by irradiation. Then it is necessary to develop reliable and suitable methods for identification and to evaluate properties of irradiated food in these countries. The most common methods for irradiated food identification are of physics and chemistry nature like thermoluminescence (TL) and microbiological load assessments in irradiated foods [13-17]. Due to the inorganic fraction present in the condiment and spices, the TL method is suitable [13,18-21] for identification of irradiated foods allowing the evaluation of the TL properties as a function of the dose response, reproducibility of TL signals, fading, and the structure of the TL glow curves due to polymineral fraction content in foodstuffs [22-29]. A significant polymineral fraction is present in Coriander (Coriandrun sativum L.) permitting to analyze its TL properties for detection purposes of previously irradiated foodstuffs. Treatment by irradiation may extend the shelf life of foodstuffs and improve product quality, it also can be used as a quarantine treatment in order to eliminate insect pests and microorganisms that are commonly present in fresh products [30-32], due to contaminated irrigation water, manure fertilized field, and infected pickers; among other sources. The microbiological load on food can be reduced by gamma irradiation and allows safe products for human consume [33-35]. The reduction of microorganisms like aerobic mesophilics colonies on the fresh condiment depends on the gamma doses delivered to the product. Coriander is a condiment of great commercial value and commonly used in Mexican dishes, but the hygienic quality can be affected mainly due to the contaminated irrigation water and deficient post-harvest handling. The irradiation treatment by gamma rays at accurate dose represent a good alternative to sterilize or disinfest the product before distribution to the consumers. The dose limit recommended by some international regulatory authorities [3,36], up to 10 kGy for dehydrated herbs, spices and seasonings [37], it is frequently used, and herbs and spices need higher doses depending on the microorganism load to control high contamination levels. In the case of fresh coriander, a dose limit of 3 kGy has a minimal effect on the volatile aromatic compounds as compared to coriander kept at cold storage [38]. Others investigations have shown that 0.45 kGy is an effective gamma dose to treat lettuce leafinternalized human pathogens [39,40]. However, the dose limits depend on the properties and bacterial pathogenic load in food as fresh-cut and vegetables [41,42]. The aim of this work is to report the main thermoluminescent characteristics of the inorganic polymineral fraction separated from fresh coriander condiment and the determination of the kinetics parameters of the polyminerals obtained by a sequential quadratic deconvolution program. Also, the dose effects on the pathogen microorganisms load present in fresh coriander as a function of gamma irradiation doses was investigated and the optimum irradiation dose to treat fresh coriander for human consumption was established.

2. Experimental procedure

The fresh coriander samples were brought from local markets in Mexico City and prepared in the laboratory. The commercial samples were washed with bi-distilled water and air-dried at room temperature. Since, the irradiation treatment of fresh coriander is not currently applied in Mexico, it was assumed the samples were not previously irradiated and later proved by TL measurements of the polymineral extracted from the samples. The polymineral separation was performed using 10 Kg of fresh coriander. For preparation of the TL experiments, each 30 g of the samples were mixed with 1000 ml of ethylic alcohol and water (70:30) solution and kept in constant stirring for 6 h in order to separate the inorganic polymineral from organic fractions. To remove the residual organic part, the polymineral fraction was washed with 30 % hydrogen peroxide followed by final washing with 10 % hydrochloric acid to eliminate the presence of carbonates in the polymineral fraction. The inorganic powder was dried with acetone and in oven at 50°C during 6 h. After that the batches were stored at room temperature in silica gel desiccators. Only 10 μ m grain size was selected to carry out the TL measurements because this size gave optimum thermoluminescent emission. To obtain the 10 μ m particles the Zimmerman method [43] applied to fine grains technique was used. The polymineral powder was analyzed by X-rays diffraction (XRD) in a Rigaku Geirgerflex D-Max diffractometer equipped with CuK_{α}-radiation (λ =1.5406 Å). X-ray patterns were obtained using 40 kV, 20 mA, and at a rate of 2 grades/min. XRD spectra of the polymineral sample showed that all samples contained quartz as the major mineral fraction of the Coriander samples. A 4 mg of powder sample for each grain size was deposited onto aluminum discs for irradiations and TL readouts. The samples were irradiated at 0.5 Gy - 15 kGy gamma doses. The 05-400 Gy dose irradiation was carried out with a Gammacell-200 (369.7 mGy/min), while for high doses (500-15 kGy) the Gammabeam 651PT irradiator (97.533 Gy/min) loaded with cobalt-60 sources was used. The irradiators were calibrated using the well known chemistry Fricke dosimeter method [44]. All samples irradiated were kept in dark condition and at room temperature (21°C), in order to avoid the effect of environmental light on TL response. A Harshaw TLD3500 reader was used for TL measurements with 2 K/s heating rate, and a constant flux of nitrogen in the chambersamples was maintained in order to reduce spurious TL signals.

For the microbiological analysis of fresh coriander, the roots were cut off and discarded. A 10 Kg of commercial

mmediately afte	er irradiation:						
Peak	Tm	Im	b	E(eV)	$s(s^{-1})$	$n_0 (cm^{-3})$	$s^* (cm^{3(b-1)}s^{-1})$
1	392.36	4.80	1.25	0.72	1.82E+08	1.26E+02	5.57E+07
2	431.60	8.80	2.00	0.92	5.69E+09	2.91E+02	1.95E+07
3	480.93	7.50	2.00	1.12	5.54E+10	2.55E+02	2.17E+08
4	526.76	3.50	2.00	1.32	4.27E+11	1.22E+02	3.51E+09
5	607.64	2.00	1.41	1.52	3.65E+11	6.53E+01	6.63E+10
6	648.41	1.40	2.00	1.72	2.02E+12	5.68E+01	3.56E+10
OM = 1.52							
After 5 days of	irradiation:						
Peak	Tm	Im	b	E(eV)	$s(s^{-1})$	$n_0 (cm^{-3})$	s^* (cm ^{3(b-1)} s ⁻¹
1	415.78	1.50	1.06	1.01	2.62E+11	295E+01	2.15E+11
2	448.67	3.54	2.00	1.21	5.64E+12	9.75E+01	5.78E+10
3	492.22	3.87	2.00	1.41	3.79E+13	1.11E+02	3.43E+11
4	533.59	2.47	2.00	1.61	2.15E+14	7.29E+01	2.96E+12
5	608.27	2.05	1.87	1.81	1.14E+14	6.71E+01	2.92E+12
6	653.97	1.00	2.00	2.01	3.38E+14	3.55E+01	9.51E+12

2500 Coriander 10 µm 2000 Intensity (a.u.) 1500 1000 500 0 10 20 30 40 50 60 70 2θ

FIGURE 1. XRD spectra of the polymineral content in commercial Coriander from Mexico City. Size particles: $10 \ \mu m$. The sharp lines belongs to the quartz.

fresh coriander was purchased from a supermarket in Mexico City. The samples were washed with bi-distilled water. Yellow and senescent leaves were discarded in order to simulate homogeneous starting conditions. Five bags containing 50 g coriander each were irradiated at 0, 0.5, 1, 5, and 10 kGy doses. Before and after irradiation the samples were handled at 12°C. Following the irradiation process, the samples were homogenized them for aerobic mesophilic microorganisms and total coliforms plate counts. Each batch sample, 50 g, was homogenized for 2 minutes in 225 ml of sterile 0.1 % wt/vol peptone water, with a Lab-blender 400 stomacher. A series of 5 samples were considered for irradiation treat-



FIGURE 2. TL Glow curves from polymineral fraction of Coriander exposed to 500-1700 Gy. The inset shows the 0-400 Gy dose range.

ments, the homogenate was then serially diluted in the same diluents. Violet red bile-MUG agar and Plate count agar (Oxoid Ltd, Basingstoke, England) were used for total coliforms and for aerobic mesophiles, respectively [45]. All media were incubated for 24 h at 35°C. Each experiment was done in two independent replicates, and four samples were analyzed for each treatment of selected irradiation dose. The aerobic plate count (APC) procedure was followed [46,47] to determine the load of microorganism in the coriander samples. All procedures were done according to the Mexican norms known as Normas Oficiales Mexicanas [48-51]. The samples were irradiated in the semi-industrial Gammabeam 651PT irradiator above mentioned.

3. Results and discussions

3.1. Thermoluminescent characteristics of polymineral fraction

The polymineral powder separated from coriander was analyzed by X-ray diffraction (XRD) and the spectra were compared with literature values [52]. Figure 1 shows the XRD spectra of the mineral sample that contained quartz as the whole component. The thermoluminescent (TL) glow curves of the samples, at different given doses, are shown in Fig. 2. The glow curves were composed of a broad bands in 273-670 K range at different gamma doses and peaked around 437-455 K. The glow curve is a result of the radiative recombination of thermally detrapped charges with a defect center and provides an identification of previously irradiated foodstuffs. The maximum peak temperature (T_m) of the glow curve shifts toward the high temperature side (Fig. 2) as the elapsed time after irradiation increases. This behavior could suggest a continuous trap distribution instead of a single trapping level responsible of the TL radiative emission. Fig. 3 shows the thermoluminescent response between 0.4-15 kGy, in the inset figure is shown the linear range between 15-150 Gy dose. This TL characteristic can be attributed to the presence of the quartz in the polymineral fraction of coriander. The reproducibility measurement of the TL signal was carried out over 12 cycles of irradiation-readout at 10 Gy. No thermal annealing was necessary after readout and subsequent irradiation, because the readout itself was enough to erase any previous TL information. A linear plot of the data gave 8 % standard deviation. For doses around 70 Gy it was possible to identify the TL of irradiated polymineral samples which was 1.15 times stronger than the TL response of nonirradiated samples exposed only to the natural background. The TL response of non-irradiated samples was similar to the response of a 60 Gy irradiated sample and due to the radiation dose contribution from natural radioactivity sources [53-55], composed mainly by uranium, thorium, and potassium. Another TL characteristic of the polymineral was the fading losses over a storage period of 30 days, in dark condition and at room temperature (296 K). The samples were irradiated at 10 kGy, a dose useful for getting a clean TL glow curve. Because the complex structure of the glow curves, composed of several overlapped peaks, it was necessary to perform a deconvolution procedure to identify the different TL peak components. Figure 4 shows the TL glow curve deconvolution at 10 kGy for the polymineral fraction of coriander. In the inset of Fig. 4 the TL fading signals after one month storage period after irradiation is illustrated. To characterized the main TL properties of the polymineral fraction, the kinetics parameters, *i.e.*, the activation energy (E) of the



FIGURE 3. TL response in the 0.4-15 kGy dose range for irradiated Coriander. The inset shows the linear 15-150 Gy dose range.



FIGURE 4. Deconvolution of the TL glow curve obtained at 10 kGy for the polymineral fraction of Coriander. The inset shows the TL fading as a function of time storage at RT.

traps, the order of the kinetics (b) and the frequency factor (s) and the pre-exponential factor (s*) were calculated (Table I). The data for deconvolution was obtained from the glow curves immediately after irradiation, and after 5 days from irradiation. A slight change of the kinetic's order b (1.25-2) was found respect to initial b (1.06-2) values and at 5 days of storage period. The values of the activation energies slightly changed between 0.72-1.72 eV and 1.01-2.01 eV, in the both cases mentioned above. The closely overlapping distribution of the deconvoluted glow peaks may be ascribed to the typical continuous traps distribution found in the polymineral fraction from foodstuffs rich in quartz [56-58]. The deconvolution was done considering the general equation and the kinetics parameters obtained using the deconvolution method based on the Sequential Quadratic Programming Glow Curve Deconvolution [59-62]:

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TABLE II. Aerobic mesophilic counts (cfu/g) of non-irradiated and
irradiated fresh Coriander samples at different gamma doses.

Dose	Experiment	Experiment	Experiment	Experiment
(kGy)	1	2	3	4
0				
(non-irradiated)	46,800,000	6,760,000	60,200,000	58,900,000
0.5	45,700	57,500	47,900	95,500
1	11,000	13,500	38,900	10,000
5	< 100	< 100	< 100	< 100
10	< 100	< 100	< 100	< 100

TABLE III. Total coliform (cfu/g) present in fresh Coriander samples.

Dose	Experiment	Experiment	Experiment	Experiment
(kGy)	1	2	3	4
0				
(non-irradiated)	871,000	692,000	257,000	100,000
0.5	302	600	300	890
1	< 100	< 100	< 100	<100
5	< 100	< 100	< 100	< 100
10	< 100	< 100	< 100	< 100

$$I(T) = sn_0 \exp\left(-\frac{E}{kT}\right) \\ \times \left[1 + \frac{s(b-1)}{\beta} \int_{T_0}^T \exp\left(-\frac{E}{kT'}\right) dT'\right]^{-\frac{b}{b-1}}$$
(1)

where n_0 is the initial concentration of the trapped charges, s is the frequency factor (s^{-1}) , b is the kinetics order, ranging from 1 to 2, and β is the heating rate (2 K/s). The input data for the deconvolution are the temperature of the peak at the maximum (T_m) , and the TL intensity at the maximum of the peak (I_m) . It was observed from the data, that unless the T_m shifts from 347 to 496 K during fading, the kinetics order did not change practically while the activation energy has a slight change to the high values. This behavior is in agreement to the theory [63,64] that predict that for deeper levels the activation energy can change to higher values respect to that corresponding to shallow traps. The increase of activation energy values on the function of the fading time is not expected from the kinetics models concerned with a single trap level, and that results can be explained considering a continuous distribution of traps. Then, during the fading for a fixed time some of the sub-levels, from the shallowest, are emptied. As the fading time increases, more deep sublevels are emptied and finally resulting in an increase of the activation energy value. From the general TL equation and

deconvolution of the TL glow curves it was possible to identify six glow peaks components from the experimental glow curves of the polymineral fraction of coriander.

3.2. Radiation effect in quality of fresh Coriander

In order to analyze the dose radiation effect on microbial load in fresh coriander, all samples were irradiated at 0, 0.5, 1, 5, 10 kGy. Five dilutions for each one were subjected to the microbiological control. The results of aerobic mesophilic bacteria (MA) are shown in Table II. Fresh coriander contained 43.2×10^6 cfu/g in average and this value was reduced to 61,650 cfu/g at 0.5 kGy dose, which is less than 50% of the limit of acceptability (150,000 cfu/g), according to Mexican regulations [50,51]. When the dose increase to 5 and 10 kGy, the mesophilic aerobic population was less than 100 cfu/g. Regarding the total coliforms (TC), the control non-irradiated samples contained 480,000 cfu/g in average, the TC were reduced to less than 100 cfu/g with a dose of 1 kGy (Table III). AM bacteria seem to be more resistant to the ionizing radiation than total coliforms present in fresh coriander. It was observed qualitatively that under our laboratory conditions, the normal green color, texture and typical flavor of the coriander leaves samples exposed to 0.5 kGy of gamma radiation, was preserved after 10 days in storage at 5°C. Our findings indicate that the irradiation treatment for coriander is an effective process for disinfecting and also to preserve the product for safe consumption.

4. Conclusions

The results obtained in this study show that the TL signal coming from the polymineral inorganic fraction separated from fresh coriander is a suitable method for detecting fresh coriander processed by irradiation. The minimum detectable dose by TL was that of 70 Gy and may be mainly attributed to quartz. The TL glow curve of extracted polymineral was composed of six overlapped TL peak components. No major changes on the kinetics parameters were found in irradiated and non-irradiates polymineral samples. Another significant result indicates that a 0.5-1.0 kGy of gamma radiation dose reduces drastically the microorganism load (99 %). The gamma dose of 1.0 kGy given to the fresh coriander assure to comply with the Mexican and international regulation on fresh food safety.

Acknowledgements

We are gratefully to DGAPA-UNAM for support the project IN121109-3 PAPIIT. The author, Brenda Ruiz Gurrola, thank also to the Academia Mexicana de Ciencias for the fellowship in the Verano de la Investigación Científica program. Also we would like to thank Dr. Pedro González from ININ for his work in the deconvolution program. Authors are grateful to Francisco García and Benjamin Leal for irradiation samples.

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