American Journal of
Food Technology

ISSN 1557-4571

Academic
Journals Inc.

www.academicjournals.com

American Journal of Food and Technology 1 (1): 66-76, 2006
ISSN 1557-4571

Oregano and Paprika Spices: Their Thermoluminescent Characteristics for Food Irradiation Dose Assessment

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Abstract: The polyminerals content from commercial dust oregano and paprika were extracted and selected by sizes of <10, 10-53, 53-74 and 74-149 μm and exposed to gamma radiation at different doses in the range 0.5-45 kGy. The glow curves from these polyminerals show an abroad TL band, centered around 450 K and composed of 9-10 glow peaks as calculated by a deconvolution procedure. The XRD analysis shows a composition of mainly quartz and feldspar, including albite, ortose and clay. The fading behavior can be mainly related to the low temperature peaks of the various minerals contained in the spices. The intensity the TL emission increases as the grain size increases for both spices. The TL properties of the polymineral contain of the spices were analyzed and it is possible to conclude that the polymineral content of both oregano and paprika can be used as efficient and practical way to determine the thermoluminescence dose assessment in herbs and spices exposed to ionizing radiation.

Key words: Food irradiation, oregano, paprika, thermoluminescence, kinetics parameters, dose-response, fading

Introduction

Thermoluminescence (TL) is a well known phenomenon exhibited by many materials (McKeever, 1985; Furetta, 2003). The interaction of the material with ionizing radiation creates electron- holes pairs and allows electrons and/or holes to be trapped in localized states inside the band gap. These trapped charges at defects in the material lattice can remain trapped for a short or long period, according to the depth energy associated to the trapping levels and to the material temperature. Subsequent heating of the irradiated crystal releases the trapped charges to later recombine themselves radioactively with the emission of light. The simultaneous recording of the intensity of the emitted light as a function of heating temperature gives rises to the well-known glow curve and its integral is proportional to the radiation dose absorbed by the irradiated sample. Therefore a thermoluminescent material may be used as a dosimeter for several applications, i.e., personal, environmental and clinical dosimetry.

Many materials, like LiF:Ti, Mg and α-Al_2O_3:C, due to their excellent TL properties, i.e., high TL efficiency, dose response, thermal stability, high sensitivity and reproducibility, are now commonly used as Thermoluminescent Dosimeters (TLD) in a great diversity fields of applications (Azorin *et al*., 1993). Some of these TLD materials are used also in dose mapping and dose assessments in standard food irradiation protocols. However, there is a need for better or more efficient

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TLD materials suitable for dose assessments in food irradiation treatments capable of processing industrials amounts of foodstuff. It is clear that a right food irradiation protocol, beside the highly sophisticated calibration procedures of typical industrial irradiators, is required in order to have a reliable *in situ* dose determination method.

Thermoluminescence observed in the natural polymineral content in irradiated herbs and spices allows a good way for dose measurements (Urbina *et al*., 1996; Urbina *et al*., 1998; Kitis *et al*., 2005). Usually, the mineral content is extracted through a chemical separation process in which the organic compounds of the herb or spice are practically non existing in the polymineral extracted sample. The polymineral is composed of minerals with high TL efficiency like, quartz, calcite and feldspars with a characteristic and reproducible TL glow curve pattern.

The aim of this research is to study the main TL characteristics of the inorganic dust extracted from paprika and oregano of Mexican origin. The results obtained show that the inorganic dust has good properties to be used as high dose dosimeter in the process of food sterilization by irradiation. Furthermore, the kinetics properties of the powder were studied using the initial rise method (IR) (Garlick and Gibson, 1948) as a first approximation for determining the activation energy and, finally, the complex structure and kinetic parameters of the glow curves have been accurately analyzed using the Computerized Glow Curve Deconvolution (CGCD) (Horowitz and Yossian, 1995; Kitis *et al*., 1998; Kitis, 2001). This research is part of a current program of food irradiation treatments carried out in the Nuclear Science Institute of the National University of México (ICN UNAM).

Materials and Methods

All samples were prepared using commercial products for domestic consumption in México. The mineral extraction was performed according to the procedure normally used in the thermoluminescence analysis in irradiation treatment of spices as reported before (Kitis *et al*., 2005). Each kind of spice was mixed with ethanol-water (60:40) solution and kept in constant agitation and centrifugation in order to obtain the fraction of mineral contaminants. To eliminate the organic residual parts, the minerals were washed with a H_2O_2 solution and distilled water; after that the mineral dust was dried with acetone. The final inorganic powder was then analyzed. The mineral content obtained by X-ray diffraction is shown in Table 1. The polymineral grains were then selected according to various grain sizes (<10, 10-53, 53-74 and 74-149 μm) using different meshes and then deposited onto aluminium discs for irradiation.

All the irradiations were performed during 2005, using the ^{60}Co irradiation facility Gammabeam 651PT (98.4 Gy/min) at the Nuclear Science Institute of UNAM. All samples were stored in dark condition and at room temperature (21°C) before and after irradiation.

TL glow curves characteristic of the samples examined were recorded using a Harshaw TL reader model 3500, with a heating rate of 2°C sec^{-1} under continuous nitrogen flux to reduce spurious TL signals. The TL signal was integrated from 21 to 400°C. The amount of sample for each experiment was 4 mg and placed onto an aluminium dish fitting to the heating strip of the TL reader. All TL measurements of the samples were running three times for each one, and the best TL signals reproducibility were chosen with not more than ±5%. The study on the kinetics parameters and the deconvolution were performed in Nuclear Physics Laboratory of the Aristotle University in Thessaloniki.

Table 1: Mineral composition of the spices

Type of spice	Minerals			
Paprika	Quartz 60%	Feldspars albite ($NaAlSi_3O_8$) 30%	Ortose ($KAlSi_3O_8$) 10%	-
Oregano	Quartz 50%	Albite ($NaAlSi_3O_8$) 20%	Calcite ($CaCO_3$) 20%	Clay 10%

Results and Discussion

TL Characteristics

The TL response as a function of the absorbed dose D, from 0.5 to 45 kGy, for paprika and oregano was investigated. Figure 1 shows the plots of TL versus D for each grain size of paprika and it is shown the linearity region of the response. Figure 2 gives the respective plots for oregano. The TL responses for both paprika and oregano are more or less spreaded. The TL response from paprika-<10 μm is the less intense and the linear region, for the various grain sizes, is extended up to 4-8 kGy, depending on the grain size. These values are in agreement with previous results obtained using European samples and the linear range was attributed to the dose response of feldspars and quartz in food (Urbina, 1998; Alvarez *et al*., 1999). The TL response from oregano is linear up to 5-10.5 kGy. For both spices the glow curves obtained in the linear range show a good defined TL peak. Table 2 lists, for both spices as well as for grain sizes, the dose levels at which saturation starts to occur.

The glow curves of all the spices studied here present a wide and intense peak centered, around 450 K. A small peak can be also observed for oregano in its high temperature region, at around 560 K. The glow curves of oregano and paprika are shown in Fig. 3 and 4, respectively. The TL glow curves in Fig. 3 and 4 are shown along with their deconvoluted components, which were calculated using a Computerized Glow Curve Deconvolution Program (CGCD), explained in detail later in the study. Nine and ten TL glow peaks were necessary for an optimum curve fitting for paprika and oregano, respectively.

The glow curve shapes are similar to those already reported in previous works (Gastélum *et al*., 2002; Calderón *et al*., 1995; Sunta and David, 1982). The polymineral extracted as inorganic part from the samples gives a glow curve which is a superposition of the glow curves of several single components. Unfired quartz (Kitis *et al*., 1990; Duller, 2003) has a low temperature peak, normally referred as the "383 K" glow peak, and a high temperature peaks around 473, 598 and 648 K. It is known that the glow curve form feldspars shows a great variety of glow peaks: the most common characteristic peaks are around 373 K, being weaker than that from quartz, a broad peak around 473 K and a high temperature peak between 573 and 673 K. Furthermore, feldspars have a higher TL efficiency and a wider dose range than that of quartz (Mejdahl, 1985; Khanlary and Townsend, 1991). Calcite presents two peaks, one at 373 K and a second one at 503 K (Randall and Wilkins, 1945). Indeed, the glow curves of both paprika and oregano should also include a low temperature TL emission related to the quartz peak and a broad TL peak centered around 423 K mainly due to feldspars.

In order to assess the stability of the TL signals, the fading was analyzed over a period of about 3 months. The samples were irradiated at different times, in steps of 8 days each, then stored in dark at RT and finally, at the end of the period of storage, all the samples were readout in only one session. This procedure is necessary to avoid any instability of the TL reader that could modify the TL emission. Figure 5 shows the fading effect in oregano for the three grain sizes of <10, 53-74 and 74-149 μm, respectively. It is observed that the TL lost, in percentage, is a little bit higher for the grain

Table 2: Grain sizes, dose levels at saturation, activation energy as determined by Initial Rise (IR) method

	Paprika		Oregano	
Grain size (μm)	Sat dose (kGy)	E (eV)	Sat. Dose (kGy)	E (eV)
<10	5	0.91	10.5	0.92
10-53	8	0.99	10	1.08
53-74	5	0.89	6	0.91
74-149	4	0.90	5	0.91
All sizes	-	0.95	-	0.99

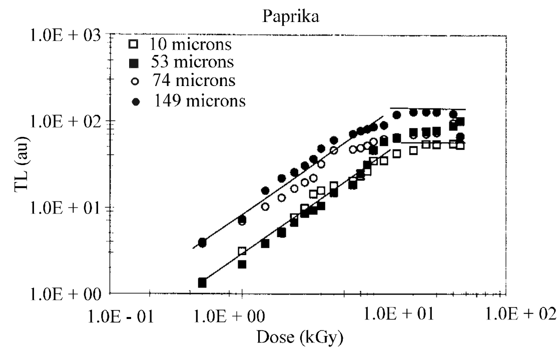

Fig. 1: TL dose response and size dependence for polyminerals extracted from Paprika

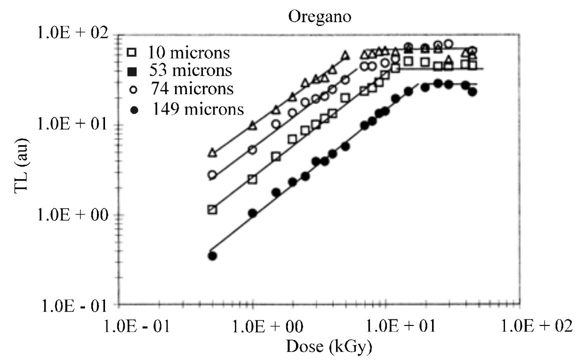

Fig. 2: TL dose response and size dependence for polyminerals extracted from Oregano

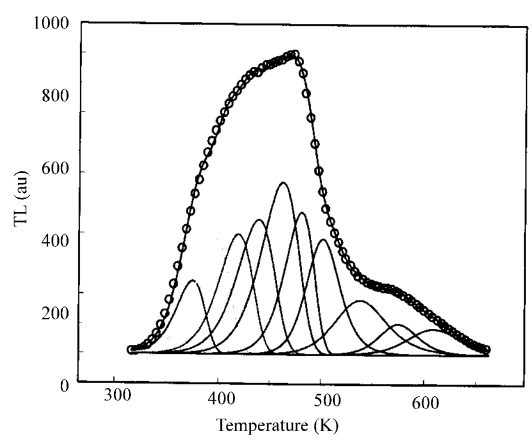

Fig. 3: TL glow curve and deconvoluted components calculated through a CGCD for Oregano. The glow curve was obtained after 10 kGy gamma exposure

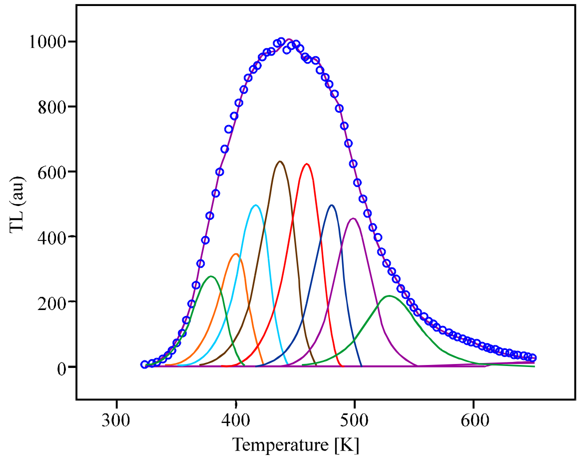

Fig. 4: TL glow curve and deconvoluted components calculated through a CGCD for Paprika. The glow curve was obtained after 10 kGy gamma exposure

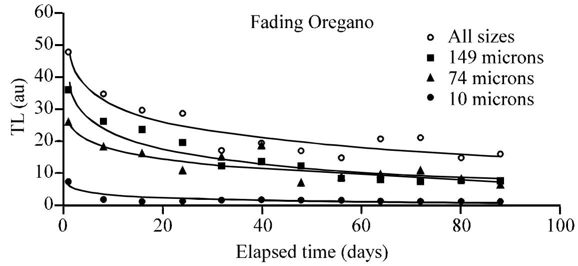

Fig. 5: Thermoluminescence fading effect polyminerals extracted from Oregano

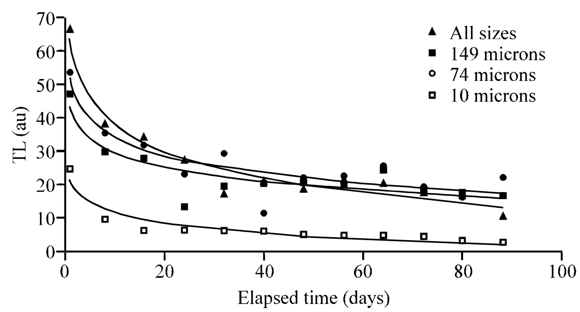

Fig. 6: Thermoluminescence fading effect polyminerals extracted from Paprika

size of 74-149 μm, about 79% at the end of the elapsed time, than for grain size of 53-74 μm, for which the TL lost at the end of the storage was only 59% of the initial value. Figure 6 shows the corresponding fading for paprika. Finally, the most relevant fading effect was detected for the grain size of <10 μm in both oregano and paprika, i.e., 86 and 89%, respectively.

Kinetics Parameters Determination

An experimental TL glow peak can be mathematically described by the Randall-Wilkins (1945) equation for first order kinetics, retrapping being absent:

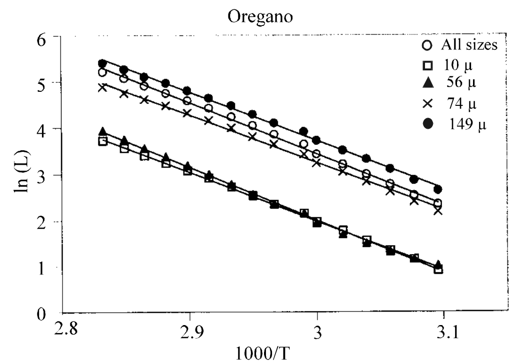

Fig. 7: Initial rise plots for the polymineral content extracted from Oregano

$$I(T) = n_0 s \exp\left(-\frac{E}{kT}\right)\exp\left[-\frac{s}{\beta}\int_{T_0}^{T}\exp\left(-\frac{E}{kT'}\right)dT'\right] \quad (1)$$

or by the Garlick-Gibson (1948) equation for the second order, when retrapping is present:

$$I(T) = \frac{n_0 s \exp\left(-\frac{E}{kT}\right)}{\left[1+\frac{s}{\beta}\int_{T_0}^{T}\exp\left(-\frac{E}{kT'}\right)dT'\right]^2} \quad (2)$$

or, by the May andPartridge (1964) general order equation, in intermediate cases:

$$I(T) = s' n_0 \exp\left(-\frac{E}{kT}\right)\left[1+\frac{s'(b-1)}{\beta}\int_{T_0}^{T}\exp\left(-\frac{E}{kT'}\right)dT'\right]^{-\frac{b}{b-1}} \quad (3)$$

where, b is the kinetics order, being $1<b\leq 2$.

A simple way for getting information about the activation energy of a peak is the well known method of the Initial Rise (IR), formerly proposed by Garlick and Gibson (1948). This method does not need any knowledge of the frequency factor as well as of the kinetics order and it is based on the analysis of the low temperature tail of a TL peak. Indeed, in this region of a peak the amount of trapped electrons can be assumed as a constant, the dependence on temperature being neglectable. In fact, with increasing temperature up to a value $T_C\leq T_M$, where T_M is the peak temperature at the maximum, (the corresponding intensity I_C should not be larger than 15% of I_M for the full TL glow-peak under evaluation) the first exponential of Eq. 1, 2 and 3 increases whereas the second term may still be unity. A further increase in temperature ($T>T_C$) makes the second term to decrease: the competition of both terms results in the maximum of I. In this assumption, as long as the second term is unity, the thermoluminescent emission can be described by:

$$I(T) \propto \exp\left(-\frac{E}{kT}\right) \quad (4)$$

The ln (I) vs 1/T plot is then made and a straight line should be obtained. From the slope, -E/k, E can be evaluated without any knowledge of the frequency factor s as well as of the kinetics order.

Figure 7 shows, as an example, the initial rise plot for Oregano. Table 2 shows the activation energy values calculated using the IR method (Furetta, 2003).

Table 3a: Kinetics parameters for "all grain sizes" of Paprika as obtained by CGCD

Peak N.	T_M (K)	E (eV)	b	s (s^{-1})
Paprika (all grain sizes)				
1	373.51	0.90	1.00	0.2086E+12
2	392.83	1.10	1.00	0.2138E+14
3	411.59	1.05	1.00	0.1038E+13
4	433.06	1.11	1.00	0.1073E+13
5	455.08	1.15	1.00	0.7019E+12
6	477.09	1.47	1.00	0.5218E+15
7	497.48	1.90	1.85	0.3040E+19
8	529.99	1.50	2.00	0.2176E+14
9	590.99	1.50	2.00	0.5805E+12

Table 3b: Kinetics parameters for Paprika (10-149 μm) as obtained by CGCD

Peak N.	T_M (K)	E (eV)	b	s (s^{-1})
Paprika (<10μm)				
1	373.33	0.84	1.00	0.3040E+11
2	393.87	1.00	1.00	0.1004E+13
3	413.85	1.01	1.00	0.2453E+12
4	434.39	1.25	1.00	0.4683E+14
5	453.38	1.28	1.00	0.2591E+14
6	473.58	1.32	1.00	0.1526E+14
7	498.14	1.94	1.81	0.7006E+19
8	531.60	1.66	1.91	0.6572E+15
9	580.33	1.50	1.99	0.1098E+13
Paprika (10-53 μm)				
1	380.00	0.90	1.00	0.1251E+12
2	400.00	1.10	1.00	0.1156E+14
3	417.02	1.12	1.00	0.5134E+13
4	437.81	1.16	1.00	0.3026E+13
5	460.00	1.31	1.00	0.3433E+14
6	480.18	1.60	1.00	0.1000E+17
7	498.75	1.90	1.75	0.2698E+19
8	530.00	1.50	2.00	0.2146E+14
9	586.96	1.50	2.00	0.7180E+12
Paprika (53-74 μm)				
1	372.01	0.85	1.00	0.5129E+11
2	394.30	1.06	1.00	0.4815E+13
3	414.75	1.00	1.00	0.1921E+12
4	437.83	1.19	1.00	0.6724E+13
5	459.30	1.19	1.00	0.1428E+13
6	481.55	1.51	1.00	0.1002E+16
7	503.25	2.13	1.81	0.4325E+21
8	537.11	1.51	1.92	0.1605E+14
9	593.30	1.50	1.93	0.5662E+12
Paprika (74-149 μm)				
1	368.03	0.86	1.00	0.9239E+11
2	388.93	1.10	1.00	0.2716E+14
3	408.08	1.05	1.00	0.1359E+13
4	429.26	1.23	1.00	0.4119E+14
5	450.75	1.15	1.00	0.9495E+12
6	475.01	1.37	1.00	0.4616E+14
7	495.75	1.72	1.70	0.5159E+17
8	530.00	1.50	2.00	0.2146E+14
9	583.51	1.50	2.00	0.8658E+12

The activation energy values of the low temperature glow-peaks, calculated by initial rise method are in good agreement with those obtained by the glow-curve deconvolution method (Table 3a, b and 4a, b).

To complete the kinetics characterization of the spices studied here, it is also necessary to use the deconvolution of the glow curve because its evident complexity. The kinetics data have been obtained using the Computerized Glow Curve Deconvolution (CGCD). The following glow peak algorithms for first and general order kinetics were used (Horowitz and Yossian, 1995; Kitis *et al*., 1998):

Table 4a: Kinetics parameters for "all grain sizes" of Oregano as obtained by CGCD

Peak N.	T_M (K)	E (eV)	b	s (s^{-1})
Oregano (all grain sizes)				
1	375.00	0.86	1.00	0.4845E+11
2	396.58	0.80	1.00	0.1752E+10
3	419.01	0.90	1.00	0.7959E+10
4	438.72	0.90	1.00	0.2584E+10
5	462.00	1.00	1.00	0.8821E+10
6	480.64	1.40	1.00	0.6752E+14
7	501.33	1.90	2.00	0.2118E+19
8	537.65	1.40	2.00	0.1420E+13
9	574.92	2.20	2.00	0.2857E+19
10	608.78	1.77	2.00	0.4719E+14

Table 4b: Kinetics parameters for Oregano (10-149 μm) as obtained by CGCD

Peak N.	T_M (K)	E (eV)	b	s (s^{-1})
Oregano (<10 μm)				
1	367.96	0.96	1.00	0.2208E+13
2	386.18	0.90	1.00	0.8725E+11
3	404.70	0.90	1.00	0.2060E+11
4	425.07	0.90	1.00	0.5481E+10
5	446.92	1.00	1.00	0.2201E+11
6	467.16	1.15	1.00	0.3349E+12
7	492.46	1.90	2.00	0.4811E+19
8	525.58	1.35	2.00	0.9846E+12
9	570.49	2.16	2.00	0.1693E+19
10	620.00	1.35	2.00	0.7117E+10
Oregano (10-53 μm)				
1	362.68	0.99	1.00	0.1063E+14
2	380.69	0.83	1.00	0.1216E+11
3	402.87	0.90	1.00	0.2338E+11
4	425.08	0.90	1.00	0.5436E+10
5	450.42	1.00	1.00	0.1772E+11
6	474.98	1.35	1.00	0.3168E+14
7	496.02	1.90	2.00	0.3135E+19
8	522.15	1.35	2.00	0.1229E+13
9	568.66	1.35	2.00	0.8865E+11
10	618.49	1.35	2.00	0.7609E+10
Oregano (53-74μm)				
1	375.00	1.00	1.00	0.4538E+13
2	392.14	1.05	1.00	0.4956E+13
3	412.42	0.90	1.00	0.1224E+11
4	436.01	0.90	1.00	0.3062E+10
5	461.97	1.21	1.00	o.1848E+13
6	482.20	1.67	1.00	0.4851E+17
7	497.84	1.90	2.00	0.2816E+19
8	526.14	1.35	2.00	0.9076E+12
9	574.98	1.36	2.00	0.6746E+11
10	619.98	1.35	2.00	0.7222E+10
Oregano (74-149 μm)				
1	365.92	0.99	1.00	0.6708E+13
2	387.13	0.80	1.00	0.3221E+10
3	411.70	0.90	1.00	0.1284E+11
4	433.38	1.03	1.00	0.1099E+12
5	452.64	1.00	1.00	0.1544E+11
6	474.97	1.23	1.00	0.1343E+13
7	494.98	1.90	2.00	0.3819E+19
8	520.00	1.40	2.00	0.4190E+13
9	563.40	2.20	2.00	0.7363E+19
10	603.20	1.40	2.00	0.4148E+11

a) For the first order

$$I(I_M, T_M, E, T) = I_M \exp\left[1 + u - \frac{T^2}{T_M^2}\exp(u)(1-\Delta) - \Delta_M\right] \quad (5)$$

where, E is the activation energy, I_M and T_M are the intensity and temperature at the peak maximum, respectively, $u = E(T-T_M)/kTT_M$, $\Delta = kT/E$ and $\Delta_M = kT_M/E$ and k the Boltzmann's constant;

b) For the general order

$$I(I_M, T_M, E, T) = I_M b^{\frac{b}{b-1}} \exp(u)\left[(b-1)(1-\Delta)\frac{T^2}{T_M^2}\exp(u) + Z_M\right]^{-\frac{b}{b-1}} \quad (6)$$

where, $Z_M = 1+(b-1)\Delta_M$

The previous Eq. 6 reduces to the second order kinetics for b = 2.

The respective frequency factors, s, can be obtained by the following expressions (Furetta, 2003):

c) First order

$$\frac{\beta E}{kT_M^2} = s\exp(-\frac{E}{kT_M}) \quad (7)$$

d) General order

$$\frac{\beta E}{kT_M^2} = [1+(b-1)\Delta_M]s\exp\left(-\frac{E}{kT_M}\right) \quad (8)$$

The curve fitting procedure was performed using the MINUIT program (James and Roos, 1997) and the goodness of the fit was tested with the Figure of Merit (FOM) (Balian and Eddy, 1977) given by:

$$FOM = \sum_{j_i}^{j_f} \frac{100\left|y_j - y(x_j)\right|}{A} \quad (9)$$

where, j_i and j_f are the first and the last channels, respectively of the region of interest, y_j and $y(x_j)$ are, respectively the TL emission and the corresponding function in the channel j, A is the integral of the TL curve in the whole region of interest. A FOM equal or less than 5% means a very good fitting.

Figure 3 and 4 show the CGCD analysis of two representative glow curves for oregano and paprika irradiated at 10 kGy, respectively. The glow curves show a very complex behavior. Table 3a, b and 4a, b give the kinetic parameters for both spices as well as for each grain size. It can be observed that the best analysis for oregano is obtained by the glow curve deconvolution in 10 peaks; for paprika this is obtained using 9 peaks. The kinetics parameters, E and s, allows to evaluate the lifetime τ (McKeever, 1985) for each trapping level: the results at RT in oregano and paprika are similar. For "all grain sizes" for paprika (E = 1.90 eV, s = 0.3040E+19 s^{-1}) and oregano (E = 1.90 eV, s = 0.2118E+19 s^{-1}), as shown in Table 3a and 4a, respectively, the values of the lifetime τ are 0.846E+6 y for paprika and 1.215E+6 y for oregano.

For less deep traps, considering 0.86 eV for paprika (Table 3b) and 0.80 eV for oregano (Table 4b), 74-149 µm grain sizes in both cases, the lifetime are 0.83 and 2.35 h, respectively. This period of time is in the range of the anomalous fading effect which appears in some feldspars causing a high loss TL, more than 50% in the first 48 h (Wintle, 1973; Visocekas, 2002; Sanderson, 1988).

A wide range of the value in the activation energy (E = 0.80, 2.20 eV) was found in oregano when the grain sizes increased. From 74-149 µm the values of the lifetime increased too: between 2.35 h and 3.816E+10 y. The behavior for paprika was quite similar. These results gives enough time to ensure that fading is not a problem in the identification between irradiated and un-irradiated paprika and oregano samples.

In general, the result from the deconvolution of the glow curve spices into several individual peaks is suggested by the various minerals contained in the materials. Each material gives a particular contribution to the total TL light which appears, then, as a superposition of multiple TL emissions. In this condition it seems physically acceptable the deconvolution into several single peaks and allows obtain the kinetics parameters.

Conclusions

The polymineral extracted by commercial dust spices like paprika and oregano has been studied using the thermoluminescence technique. Various TL properties have been investigated and the results concerning the TL response as a function of the delivered dose, fading, analysis of the TL glow curve and the kinetics parameters are reported. In general, the extracted polymineral content of both spices seems could be potentially used for dose assessment during herbs and spice irradiation treatments.

The particle size has a strong impact over the TL emission and dosimetric purposes; it is evident the necessity to use the largest possible grain size for getting the highest TL sensitivity. On the other hand, the lowest grain size should allow obtaining a better homogeneity between the various materials listed in Table 1. In the case of coarse grains it is very possible to have high variation in both glow-curve shape and sensitivity due to different portions of each material in different samples. Because the fading effect is always high, it is necessary to read out the irradiated samples immediately after irradiation or to use a determined elapsed time after irradiation exposure: it this way no corrections have to be applied for the dose determination. In any case, an accurate study on fading is in progress to check if the dusts present any anomalous fading effect, i.e., fading concerning to the high temperature side of the glow curve.

Acknowledgements

This research has been partially supported by Oficina de Colaboración Interinstitutional of UNAM and Intercambio Académico from Universidad de Sonora. We would like to thanks Mr. Martín Cruz and José Rangel for their help in the samples irradiation and for assisting in computing control of the irradiator facility at the Unidad de Irradiación y Seguridad Radiológica of ICN UNAM.

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