Thermoluminescent characteristics of LiF:Mg,Cu,P and CaSO₄:Dy for low dose measurement

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Thermoluminescence (TL) characteristics for LiF:Mg,Cu,P, and CaSO₄:Dy under the homogeneous field of X-ray beams of diagnostic irradiation and its verification using thermoluminescence dosimetry is presented. The irradiation were performed utilizing a conventional X-ray equipment installed at the Hospital Juárez Norte de México. Different thermoluminescence characteristics of two materials were studied, such as batch homogeneity, glow curve, linearity, detection threshold, reproducibility, relative sensitivity and fading. Materials were calibrated in terms of absorbed dose to the standard calibration distance and they were positioned in a generic phantom. The dose analysis, verification and comparison with the measurements obtained by the TLD-100 were performed. Results indicate that the dosimetric peak appears at 202°C and 277.5°C for LiF:Mg,Cu,P and CaSO₄:Dy, respectively. TL response as a function of X-ray dose showed a linearity behavior in the very low dose range for all materials. However, the TLD-100 is not accurate for measurements below 4mGy. CaSO₄:Dy is 80% more sensitive than TLD-100 and it show the lowest detection threshold, whereas LiF:Mg,Cu,P is 60% more sensitive than TLD-100. All material showed very good repeatability. Fading for a period of one month at room temperature showed low fading LiF:Mg,Cu,P, medium and high for TLD-100 and CaSO₄:Dy. The results suggest that CaSO₄:Dy and LiF:Mg,Cu,P are suitable for measurements at low doses used in radiodiagnostics.

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I. Introduction

Medical applications of ionizing radiation are the most important sources irradiation of the population. Ionizing radiation is used in medicine in three areas: nuclear medicine, radiotherapy and diagnostic radiology, the latter use X-ray equipment to obtain images of the inside of the patient’s body.

Dosimetric investigations in diagnostic radiology have been increasing in importance in the last two decades. The most widely used technique in radiation dosimetry is thermoluminescence (TL).

Several types of thermoluminescent dosimeters (TLD) are commercially available for a wide range of applications: personnel, environment and medical dosimetry, nuclear accidents, etc.

Lithium fluoride doped with magnesium and titanium, known commercially as TLD-100, is still the most commonly used radiation dosimeter. It has become popular because of several properties, such as tissue equivalence, relative low fading and the possibility to manufacture the material with acceptable reproducibility [1], [2], [3], [4].

TLD-100 has some features that do not entirely suit for use in low dose X-ray such as low sensitivity (which is why it is necessary to calibrate every use), poor detection threshold and disagreement in several reports about the fading [5], [6], [7]. In this work this material was used only as a reference dosimeter.

Other material with nearly tissue equivalence is lithium fluoride doped with magnesium, copper, and phosphorus (LiF:Mg,Cu,P) [8] and [9]. It has several important advantages compared to TLD-100 such as higher sensitivity, low fading, good detection threshold; however, this has not yet been proposed for routinely use for dosimetric applications as the TLD-100.

On the other hand, there are materials over-respond due to their higher effective atomic number Z. Thus, they have higher sensitivity and are characterized as non-tissue equivalent materials. This materials are calcium sulfate (CaSO₄) and calcium fluoride (CaF₂) among others and are used for environmental monitoring.

For environmental monitoring of conventional medical installations, dosimeters should be placed at least a month, so its fading in this period of time should be the minimum. Calcium fluoride presents a rapid fading in a short period of time [10], [11], therefore this materials are not suitable for routine monitoring of low dose X-ray.

Calcium sulfate has advantages for environmental TLD. It can easily be prepared, is $\approx 30$ times as sensitive as TLD-100, and exhibits considerably less fading than calcium fluoride [12]. However this characteristics have been realized for indoor dosimetry of gamma radiation areas in around of the nuclear installations in the worldwide [13], [14], [15]. But not there are measure-
ments of environmental X radiation delivered in medical installations.

The aim of this study is to determine the dosimetric characteristic of two different thermoluminescent materials LiF: Mg, Cu, P and pellets synthesized in Mexico based on CaSO$_4$:Dy, and compared them with TLD-100 for low dose X-rays used in diagnostic radiology in similar conditions or close to real working conditions which may reduce the uncertainties associated with commercial TLDs.

II. Materials and Methods

A. Materials

The materials and equipment used are listed below:

- TL dosimeters
  - 18 chips of TLD-100 of 3 x 3 mm and 1 mm thick. (Harshaw Chemical Company, Solon, OH, USA).
  - 20 discs of 3 mm diameter and 1 mm thick of CaSO$_4$:Dy (CICATA-Legaria, IPN).
  - 16 discs of 4.5 mm diameter and 1 mm thick of LiF:Mg,Cu,P (MIKROLAB s.c., Poland).
- Harshaw Thermo Scientific TLD Reader (Model 3500).
- Harshaw Thermolyne heating muffle (Model 1400).
- Radcal Accu-Gold+ multisensor (Model AGMS-D).
- Conventional X-ray equipment (CMR model MRH-II E GMX 325AF SBV-1 with Rotating Anode X-ray tube).

B. Methodology

Dosimeters received a standard annealing treatment before exposure to radiation. Depending on the type of material, thermal annealing schemes were: 240°C for 10 minutes to LiF:Mg,Cu,P and 300°C for 30 minutes to CaSO$_4$:Dy and 400°C for 1 hour followed by 100°C for 2 hours to TLD-100. The method of slow cooling inside the muffle was used to reach room temperature for all cases.

The radiation was performed with a conventional X-ray equipment of radiology area at Hospital México specifically designed for general radiographic procedures.

The readings of the TL materials are performed in a reader. The reading cycles were varied depending on the material as shown in Table I. In order to eliminate the contribution by triboluminescence all readings were performed in an atmosphere of high purity N$_2$.

| Parameters                     | LiF:Mg, Cu, P | LiF:Mg, Ti CaSO$_4$:Dy |
|-------------------------------|---------------|------------------------|
| Preheating temperature        | 100°C         | 50°C                   |
| Preheating time               | 12 s          | 5 s                    |
| Preheating speed              | 8°C/s         | 10°C/s                 |
| Max. Heating temperature      | 240°C         | 350°C                  |
| Acquisition time              | 20 s          | 30 s                   |
| Annealing temperature         | 240°C         | 350°C                  |

TABLE I. Reading parameters for TLD materials

1. Batch homogeneity

Batches of the three materials already described were annealed with the parameters listed above according to each type of material. They were then irradiated with a conventional X-ray equipment at a dose of 5 mGy (considering their possible use for clinical dosimetry) for the TLD-100 and LiF:Mg,Cu,P whereas the CaSO$_4$:Dy was irradiated at a dose 1 mGy (for applications in environmental dosimetry and personal).

Immediately after irradiation, the materials were read under the parameters mentioned above, the TL emission values were recorded as $M_i$, for $i = 1, 2, 3, ..., N$. Later they were erased and re-read to determine the background reading or reading to a zero dose and these values were recorded as $M_0$. The average values of these readings was calculated and expressed in terms of:

$$\Delta_{max} = \frac{(M - M_0)_{max} - (M - M_0)_{min}}{(M - M_0)_{min}} \times 100\%$$

where $\Delta_{max}$ is the index of uniformity for given batch and must be less than or equal to 30% in order to be considered acceptable values [10].

2. Glow curve

To study the TL curves, 6 dosimeters were used, which were previously annealed, they were protected from light and irradiated to 80 kVp, 300 mA, with an exposure time of 0.5 s, which means a dose of 14.69 mGy. We used a focus-surface distance (FSD) of 80 cm and a field of 10 x 10 cm$^2$. Readings were taken at 24 hours post irradiation.

3. TL response as function of dose (Linearity)

A solid state multisensor was used for calibration of TL materials in terms of absorbed dose. 16 dosimeters of each material were divided into groups of 4 and dosimeters were placed into capsules of latex, which are arranged adjacent to the AGMS. These were irradiated with the equipment of conventional radiology under the following parameters: 80 kVp, 300 mA to a DFS 100 cm, with
FIG. 1. Glow curve at a dose of 14.6mGy for TLD-100 and CaSO$_4$:Dy. Note that the thermoluminescent intensity of TLD-100 is multiplied by 50.

a size of 10 × 10cm$^2$ to different values of the product of current-time 18mAs, 36mAs, 75mAs, 150mAs, which gave the 1.75mGy, 3.52mGy, 7.29mGy and 14.69mGy doses, respectively. Dosimeters were readed at 24 hours post-irradiation. In order to obtain the TL response as a function of the radiation dose for each of the materials, the TL intensities were plotted versus the obtained from AGMS in the range of doses studied.

4. Detection threshold, $D_{LDL}$

The lower detection limit is defined as the lowest dose that can be detected with an acceptable confidence level $[17]$, which is defined as 3 times the standard deviation of the reading at zero dose, and is expressed in units of absorbed dose.

Four dosimeters of each material previously annealed as indicated in $[11B]$ were irradiated at a dose of 11.49mGy for TLD-100 and 7.29mGy for LiF:Mg,Cu,P, and CaSO$_4$:Dy with the conventional X-ray equipment described above, they were readed at 24 hours after irradiation and an equal cycle is performed again. Detection thresholds for the three materials were calculated from the following expression:

$$D_{LDL} = 3\sigma_{BKG} \times \Phi_C,$$

where $\sigma_{BKG}$ is the standard deviation at zero dose and $\Phi_C = \frac{D}{M}$ is the calibration factor for determinated dose $D$.

5. Repeatability

In order to study the repeatability of the material at low doses, a total of six dosimeters, two of each type were used. The test was performed for ten consecutive cycles, i.e., thermal annealing treatment, irradiation and reading with the same conditions for each annealing cycle. Annealing was conducted according to the conditions mentioned in Section $[11B]$ the irradiation was performed at a dose of 5mGy and readings were made 24 hours post-irradiation using the same parameters mentioned in section $[11B]$. The repeatability of the TL response as a function of absorbed dose was calculated with the following expression:

$$R = \frac{100\sigma}{\bar{x}} \leq 7.5\%,$$

where $\sigma$ is the standard deviation and $\bar{x}$ is the average of all readings during the 10 cycles.

6. Relative sensitivity

The relative sensitivity of each material was determined by comparing the mean values obtained in the reproducibility test, considering TLD-100 as a reference.

7. Fading at room temperature

Fading of dosimeters as a function of time was studied. To do this, 12 dosimeters each type of material were used, previously annealed, then they were irradiated at a dose of 14mGy and they kept it stored all the time at a temperature of 20°C. Readings were taken at the following post-irradiation time: 3h, 24h, 48h, 120h, 168h, 288h, 720h (1 month).

III. Results and discussion

In Figures $[1]$ and $[2]$ are shown TL glow curves obtained for the three different materials at low doses of X-rays.
used in the field of radiology.

The dosimeter LiF:Mg,Ti has a spectrum with four peaks centered at temperatures of 155°C, 192°C, 243°C and 305°C; CaSO₄:Dy has two peaks centered at 179°C and 277°C. Finally, LiF:Mg,Cu,P presented three peaks that appeared at 148°C, 202°C and 237°C. Dosimetric peaks for each material are centered to the following temperatures $T = 243°C$, $T = 277°C$ and $T = 236°C$, respectively.

In Table II shows the values of TL response of different materials for the range of 1.76$mGy$ to 14.69$mGy$ during calibration or response in a dose dependent. It was obtained a variation in the relative standard deviation of the TL readings from 0.09 to 0.32 for the TLD-100, 0.02 to 0.22 for CaSO₄:Dy and 0.03 to 0.2 for LiF:Mg,Cu,P. It was observed that the standard deviation was very high at below 4$mGy$ dose mainly for the TLD-100 (32%). For this reason, many reports also analyze just over 5$mGy$ doses [18], [19].

| Exposition time [s] | AGMS [mGy] | TLD-100 [nC] | CaSO₄:Dy [nC] | LiF:Mg,Cu,P [nC] |
|---------------------|-------------|--------------|---------------|-----------------|
| 0.06                | 1.758       | $\bar{x} = 42.9, \sigma = 32\%$ | $\bar{x} = 1201.2, \sigma = 22\%$ | $\bar{x} = 1127.0, \sigma = 8\%$ |
| 0.12                | 3.521       | $\bar{x} = 68.6, \sigma = 20\%$ | $\bar{x} = 2461.5, \sigma = 16\%$ | $\bar{x} = 2314.5, \sigma = 5\%$ |
| 0.25                | 7.291       | $\bar{x} = 115.4, \sigma = 10\%$ | $\bar{x} = 5064.0, \sigma = 17\%$ | $\bar{x} = 4727.5, \sigma = 3\%$ |
| 0.5                 | 14.694      | $\bar{x} = 230.1, \sigma = 9\%$ | $\bar{x} = 10395.0, \sigma = 2\%$ | $\bar{x} = 9113.0, \sigma = 20\%$ |

TABLE II. Values obtained during calibration of the materials to low dose X-rays.

| TL material | Repeatability | Detection threshold [µGy] | Relative Sensitivity |
|-------------|---------------|----------------------------|---------------------|
| TLD-100     | 3.18          | 160                        | 1                   |
| CaSO₄:Dy    | 3.20          | 6                          | 82.3                |
| LiF:Mg,Cu,P | 4.00          | 12                         | 66.3                |

TABLE III. Repeatability, detection threshold and relative sensitivity of different TLDs in a diagnostic X-ray beam.

Figure 3 shows the the dose-response curve for an 80$kVp$ X-ray in the low dose in log-log scale. A linear plot in the log-log scale with the slope equal to 1 indicates a linear dose response. The error bars in the graph corresponds to 5%. Non-linearity, as reported by some authors [20], [21] was observed for the TLD-100 below 4$mGy$. These findings are important and should be made available to physicians and occupationally exposed using TLD-100 for the monitoring of low and very low doses.

The values for repeatability test for each type of material followed by 10 cycles of irradiation, Eq. (2), are presented in Table III. Also in Table III it is shown the detection threshold and relative sensitivity for the TLD-100, LiF:Mg,Cu,P and CaSO₄:Dy.

The three materials showed values below 7.5%, i.e., 3.18% for TLD-100, 3.20% for CaSO₄:Dy and 4.0% for the LiF:Mg,Cu,P. In general the three materials showed very good repeatability for low dose X-ray. In Figure 4 the values for all materials are presented and include error bar of 5%.

CaSO₄:Dy is about 82% more sensitivity than TLD-100, while the LiF:Mg,Cu,P was about 66% higher than the TLD-100. In other investigations [19], [22], [23] have been reported sensitivity factor values about 60% more than TLD-100 for CaSO₄:Dy. The most sensitive is CaSO₄:Dy and the second one is LiF:Mg,Cu,P. Among all materials the TLD-100 presented the lowest TL sensitivity.

It is not easy to compare sensitivity values obtained
FIG. 4. Repeatability test for LiF:Mg, Cu, P and CaSO₄:Dy compared to the TLD-100 after 10 cycles of irradiation. Note the values of the TLD-100 are multiplied by 50.

in this study with those from the literature, because this parameter varies significantly with beam energy. Most frequently, sensitivity values are presented for the ⁶⁰Co energy, which are not useful for dosimetry purposes in low energy beams. The results obtained showed that, in the diagnostic radiology energy range, the differences in sensitivity among the materials are even more accentuated than for the high energy beam of ⁶⁰Co.

Typical TL reproducibility values are between 2% and 10% [24], [25], [6], [26]. The results of this study were all within the expected range. Moreover, all materials, presented very good performance, with reproducibility values below 4%.

FIG. 5. Graphical representation of the evolution of the glow curves for TLD-100 from 3 hours (Note 24h) up to 1 month (note 720h) after irradiation.

From the three materials, LiF:Mg,Cu,P presents less reproducibility, reading up to 240°C was not enough to annealing. Specifically, there is a distribution of traps associated with low intensity peaks located between 270°C and 300°C that are not annealed [27]. For this reason, upon reading residual signals remain dependent on their dosimetric history, so after a certain time measurement reproducibility is impoverished.

In Figures 5, 6 and 7, the fading of peaks of glow curves for the three materials are shown in a period of one month.

It is observed clearly the slight decrease in the intensity of the dosimetric peak for each of the materials, the first peak had a higher fading, completely disappearing at the 288h for the TLD-100 and CaSO₄:Dy and 720h (1 month) for LiF:Mg,Cu,P.

FIG. 6. Graphical representation of the evolution of the glow curves for CaSO₄:Dy from 3 hours (note 24h) up to 1 month (note 720h) after irradiation.

The above mentioned figures [5, 6, and 7] show a small shift to higher temperature are seen in the peak temperature of the main dosimetry peak. It is known that a glow peak with kinetic order greater than one (non-first-order TL glow peak) shifts to higher temperatures with decreasing the population of trapping states. Storing the TL dosimeter causes depopulation of trapping states due to fading. Therefore, the TL glow peaks shift to higher temperature with increase in storage time.

FIG. 7. Graphical representation of the evolution of the glow curves for LiF:Mg,Cu,P from 3 hours (note 24h) up to 1 month (note 720h) after irradiation.

By analyzing the relative intensities (as shown in Figure 8), obtained for each measurement, the TLD-100 showed a slow fading (11%) for a period of 3 hours to 48 hours, after this the fading was 15% between 48h and 720h. In the case of CaSO₄:Dy the fading was 11% between 3h and 48h and 35% from 48h up to 720h presenting the greatest fading compared to other materials. Finally, LiF:Mg,Cu,P presented a fading of 7% between
3h and 48h and slow fading (8.8%) from 48h until 720h post irradiation.

The CaSO$_4$:Dy was the material with a higher fading in the period of one month at room temperature, which is in agreement with that reported in the literature [28], [29], in the case TLD-100 showed a high fading as reported by [30] and [31], while the LiF:Mg,Cu,P dosimeter was experienced slower fading due to loss of some of the initially trapped charges, between irradiation and reading the influence of heat (even at room temperature, thermal fading) or exposure to unwanted light (optical fading). It is further known that the response of LiF:Mg,Cu,P is more stable at ambient temperature than the TLD-100 [32], [33].

IV. Conclusions

In this research, thermoluminescence materials LiF:Mg,Cu, P and CaSO$_4$:Dy were characterized to low doses of X-rays, which correspond to radiological diagnosis by the following dosimetric tests: homogeneity batch reproducibility, sensitive factor, detection threshold, linearity and fading. The experiments were carried out simultaneously with the tests for TLD-100, so that the results are directly comparable.

The materials have a linear behavior for the range of doses studied (CaSO$_4$:Dy and LiF:Mg,Cu,P). TL reading for TLD-100 have a high uncertainty below 4mGy so we conclude that is not precise and it has a non-linear behavior in the dose range described above. This finding are very important and should be made available to researchers and medical practitioners that they use TLD-100 dosimeters for low dose measurement in diagnostic radiology.

The CaSO$_4$:Dy is the material with higher threshold detection with very high sensitivity to low doses X-rays so we suggest its use in environmental and personal dosimetry in diagnostic radiology.

LiF:Mg,Cu,P has high sensitivity and excellent dosimetric characteristics better than the TLD-100, and is the material that show fewer fading in natural light and environmental conditions. We suggest its use for monitoring both environmental and occupational doses in rooms with low doses of radiation.

Finally we conclude that the system formed by the CaSO$_4$:Dy and LiF:Mg,Cu,P is effective for detection of very low doses delivered by X-ray equipment. This system is very useful to physiologists, medical physics, occupationally exposed workers, etc. Our final suggestion is that in every area of health where work with ionizing radiation combined use of both materials becomes necessary to achieve a comprehensive dosimetry monitoring.

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