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A comparative study of the thermoluminescence properties of several varieties of Brazilian natural quartz

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ABSTRACT

The following varieties of natural quartz, as the blue, the green, the red, the pink, the black, the sulphurous and the milky quartz, have been investigated concerning their thermoluminescence properties. For comparison sake natural colorless alpha quartz has been include. Since X-rays diffraction analysis has shown that all of them have the same crystal structure as the alpha quartz, it is expected that no great change in the TL property should be found, however, that was not the case. The TL peaks at 110, 175, 220, 325 and 375 °C observed in the alpha quartz are not found in all the varieties of quartz, for instance, the sulphurous quartz presented only 110° and 245° peaks, the pink one presented just 110, 220 and 375 °C peaks and so on. In respect to TL response as function of gamma ray dose a quite varied behavior has been observed and discussed.

1. Introduction

Due to its important applications in high technology, industry, jewelry and archaeological dating, quartz has received considerable attention and extensive work has been carried out on different aspects like color centers, thermoluminescence, EPR, etc. Quartz is also known as rocky crystal and was studied by Strabo as early as 64 B.C. [3]. Colored quartz such as amethyst and citrine always attracted people's attention. Goldschmid [4] was one of first workers to carry out a study on thermoluminescence of natural quartz. The usefulness of thermoluminescence of quartz in archaeological and geological dating technique stimulated further such investigations [8,13,14,1,17,9].

It is quite accepted that the glow curve of a natural alpha quartz presents peaks at 110 (150), 220 and 325 or 375 °C above room temperature. In the literature one finds 110 °C peak occurring between 90 and 130 °C and the peak at 250 °C is reported by some author rather than being observed at 220 °C. Furthermore, McKeever [10] has pointed out that the glow curves show a variety of shapes depending on the nature and origin of quartz.

Chawla and Singhvi [2] have shown that the TL growth curve in the dose range about 1000 Gy is composed of three parts: (1) linear up to about 100 Gy, (2) almost constant between 100 and 400 Gy, (3) a fast growth above 400 Gy. Watanabe et al., [22] have investigated the 325 °C TL peak intensity as a function of dose in quartz grains extracted from sediments and have shown that the TL growth occurs due to two contributions-, the first one due to thermodynamically created oxygen vacancies and the second to oxygen vacancies generated by radiation. In both cases oxygen vacancies capture electrons liberated by radiation. Toyoda and Ikeya [20] have shown that such oxygen vacancies capture two electrons and release electron between 120 and 300 °C, generating E'$_1$-center. The remaining electron is liberated in temperature region of 300 to 450 °C. The liberated electrons give rise to 220 and 325 °C TL peaks. The model proposed by Watanabe et al. (1998/2008) is based on the Mitchell et al. [12] model for *F*-center formation in KCl. The mathematical expressions have been modified by [21] to conform to physical conditions.

In the present work, thermoluminescence properties of seven varieties of natural quartz have been investigated. Alpha and synthetic quartz have been added for comparison.

2. Materials and experiments

The seven varieties of natural Brazilian quartz here investigated are: the blue, the green, the milky (or white), the sulphurous, the pink (or rose), the red and black quartz. Their crystal structures have been analyzed using the XRD technique. Since they are natural minerals they contain several foreign elements and their concentrations were determined by X-rays fluorescence and by ICP-MS techniques.

The samples to be investigated were pulverized and sieved to retain grain sizes between 0.08 and 0.180 mm. They were annealed at 600 °C for 30 min to eliminate previous radiation effects. The irradiation has been carried out at the Institute of Energy and Nuclear Researches using ^{60}Co-source. The glow

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curves of samples irradiated until 10.000 Gy were made by the Daybreak model 1100 TL reader. Heating rate of 4 °C/s has been used in all the experiments. The TL emission spectra have been recorded in a TL reader using a Hamamatsu 551S PMT and UNICRON100- FUNBEC monochromator. The spectra were obtained in the temperature range from 225 to 450 °C.

3. Results and comments

X-rays diffraction measurements show that different varieties of quartz have similar pattern to standard alpha quartz indicating they have same crystal structure of alpha quartz. Table 1 lists the major impurities as found by the ICP-MS technique.

The green quartz, also called prasiolite or greened-amethyst is produced when amethyst from certain localities, is heated between 300 and 600 °C ([19,15,16]. The pink or rose quartz occurs in massive deposits and rarely as isolated crystals. [5,6] has shown that the pink color is due to Mn^{3+}. In the natural blue quartz the color is due to scattering of incident light by minute inclusions, [7]. If the light can be reflected off the surface of a thin slab, the blue color is observed. When the same slab is held so that the transmitted light can be observed, the blue color is replaced by orangish-brown color.

The TL glow curves of varieties of quartz are shown in Figs. 1 and 2. Since 110 °C peak is very short lived, a pre-heating at 110 °C was performed in all the varieties, except in the synthetic quartz, before TL read-out. The glow curves are registered after laboratory

Table 1
Impurities (in weight %) in nine varieties of quartz. Only the impurities present in high concentration are listed.

	Alpha	Blue	Green	Black	Pink	Red	Sulphurous	Milky	Artificial
SiO_2	99.4	97.97	98.1	97.34	98.83	98.35	99.17	98.58	99.2
Al_2O_3	0.22	0.49	0.82	0.3	0.26	0.30	0.07	0.10	0.16
MnO	0.002	0.004	*d.*l.	0.02	0.002	0.004	0.003	0.140	*d.*l.
MgO	0.01	0.01	*d.*l.	0.01	0.01	0.01	0.01	0.01	*d.*l.
CaO	0.05	0.01	0.03	0.19	0.04	0.07	0.01	0.01	*d.*l.
Na_2O	0.02	0.02	*d.*l.	0.02	0.02	0.02	0.02	0.02	0.10
K_2O	0.01	0.13	0.73	0.1	0.01	0.03	0.01	0.01	0.03
TiO_2	0.002	0.241	0.06	0.008	0.005	0.007	0.002	0.001	*d.*l.
P_2O_5	0.01	0.013	0.01	0.064	0.01	0.005	0.003	0.003	*d.*l.
Fe_2O_3	0.02	0.58	0.03	0.18	0.03	0.69	0.01	0.01	0.13

*d.*l.= determination limits ($\leq$ 0.001).

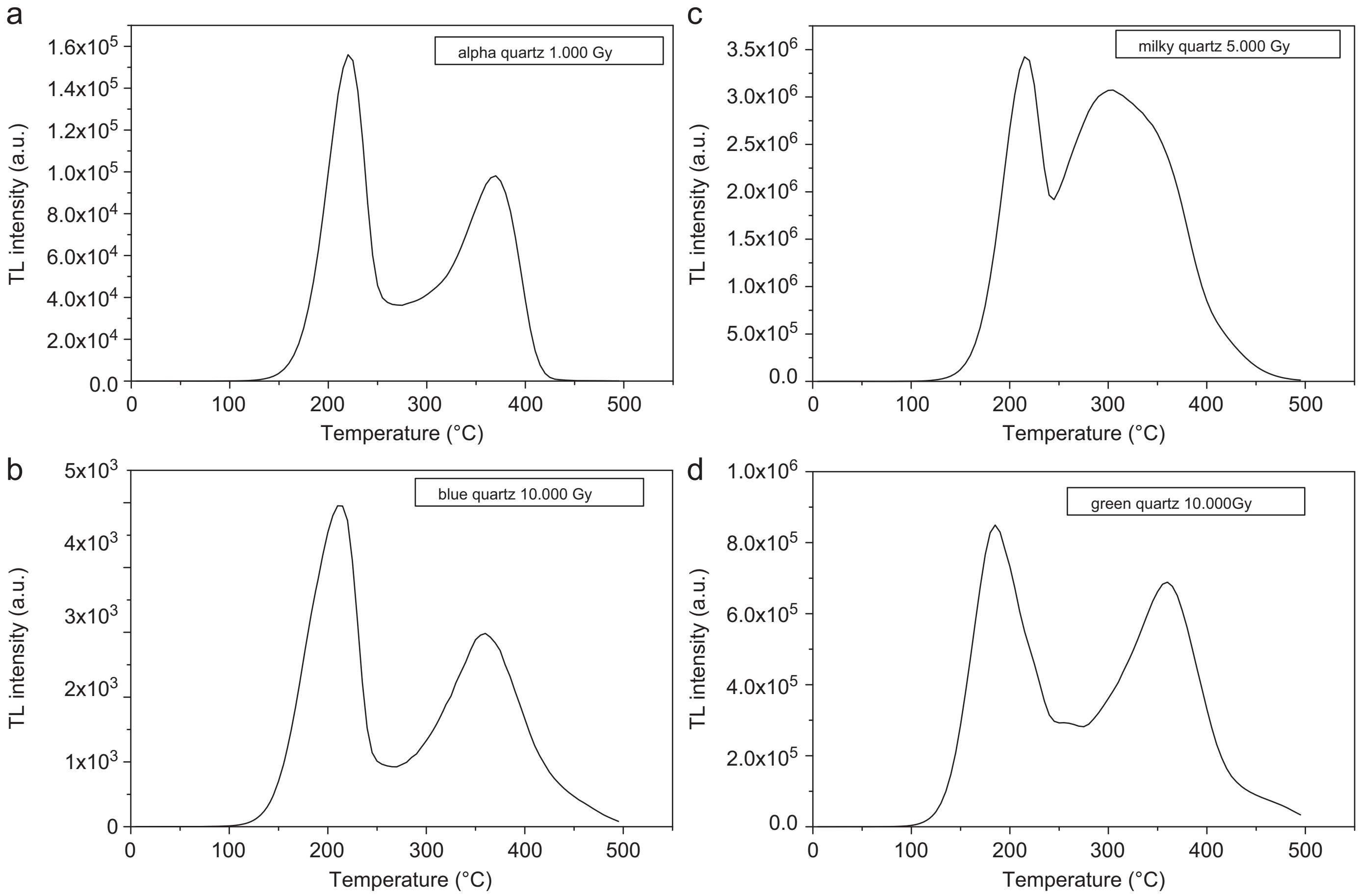

Fig. 1. (a) Glow curve of alpha quartz. (b) Glow curve of blue quartz. (c) Glow curve of milky quartz. (d) Glow curve of green quartz. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

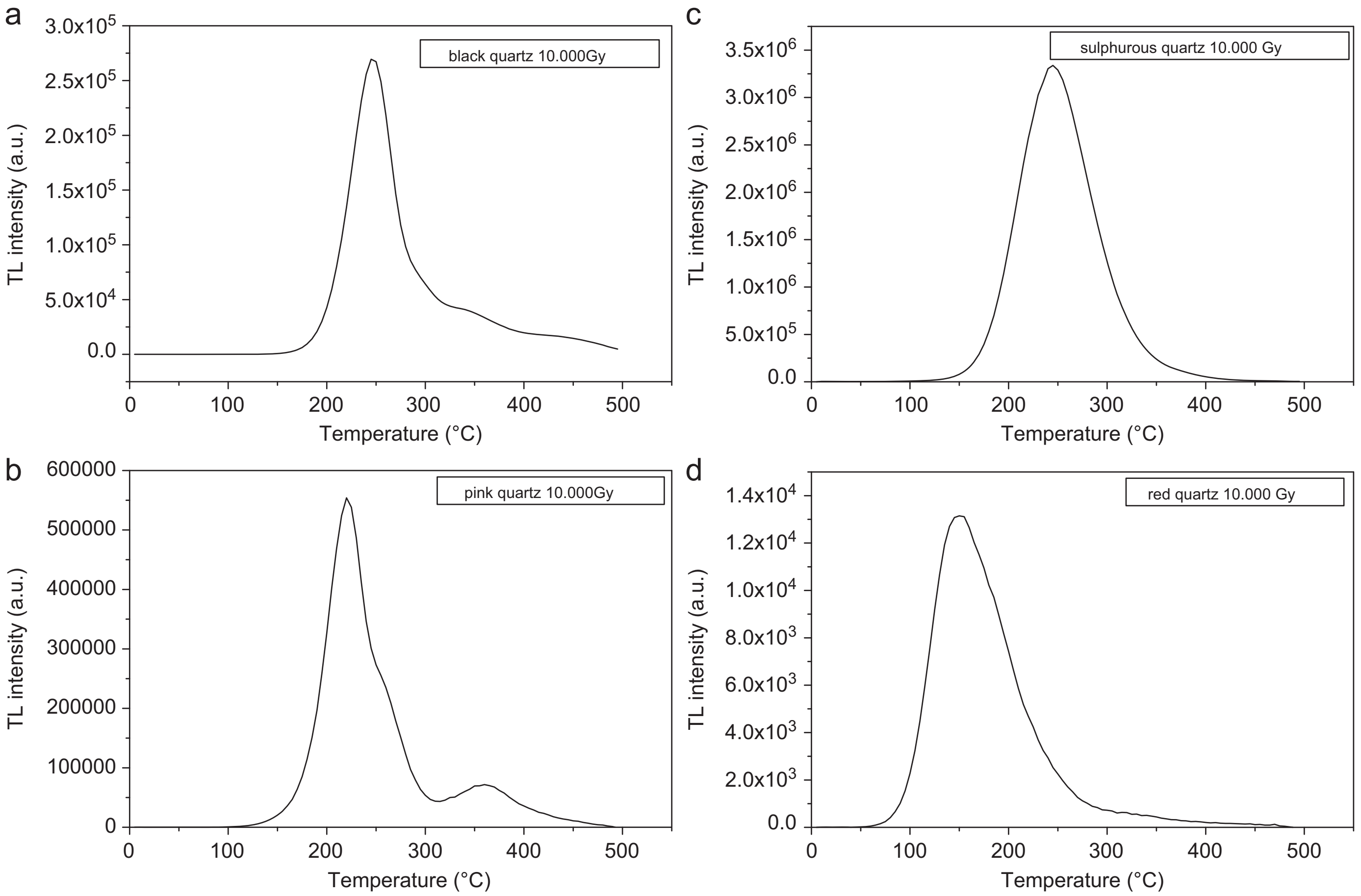

Fig. 2. (a) Glow curve of black quartz. (b) Glow curve of pink quartz. (c) Glow curve of sulphurous quartz. (d) Glow curve of red quartz. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

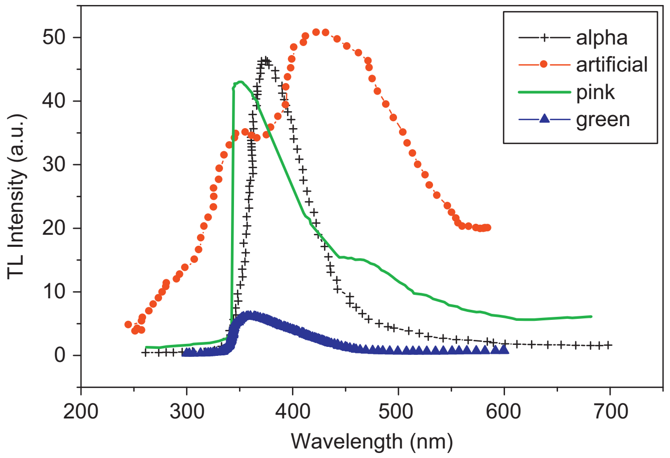

Fig. 3. TL emission spectra of alpha, artificial, pink and green quartz. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

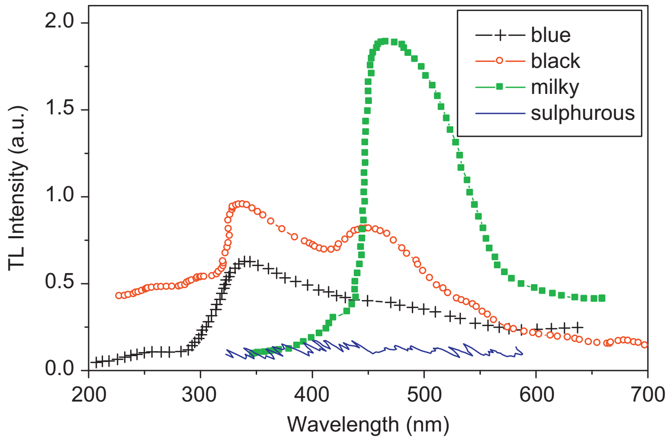

Fig. 4. TL emission spectra of blue, black, milky and sulphurous quartz. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

irradiation with doses ranging from 1 up to 10 kGy, except for the alpha and the synthetic quartz which the maximum irradiation was limited to 1 kGy.

First, it was observed that only the blue quartz presented a glow curve, not equal, but similar of the alpha quartz while all the others differed, some of them greatly. Actually, as already mentioned by [10] the glow curves of alpha quartz show an enormous variety of shapes. Up to now no clear general explanation has been found, although it is known that high dose irradiation and high temperature annealing do affect the position and the height of the peaks. In the present work as Figs. 1(a)–(d) to 2(a)–(c) show as the common aspect there is the fact that the peak around 220 °C is observed in all the varieties of quartz. Actually, in the alpha, in the milky, in the black, in the sulphurous and in the pink quartz the peak occurs at 220–230 °C, while in the blue and in the red quartz it shifts to 180–190 °C. The TL peaks at 325 and 375 °C are typical in alpha quartz; they are observed in the blue, in the milky and in the green quartz; in the black quartz they appear very weakly, while in the pink one no 325 °C peak is observed and

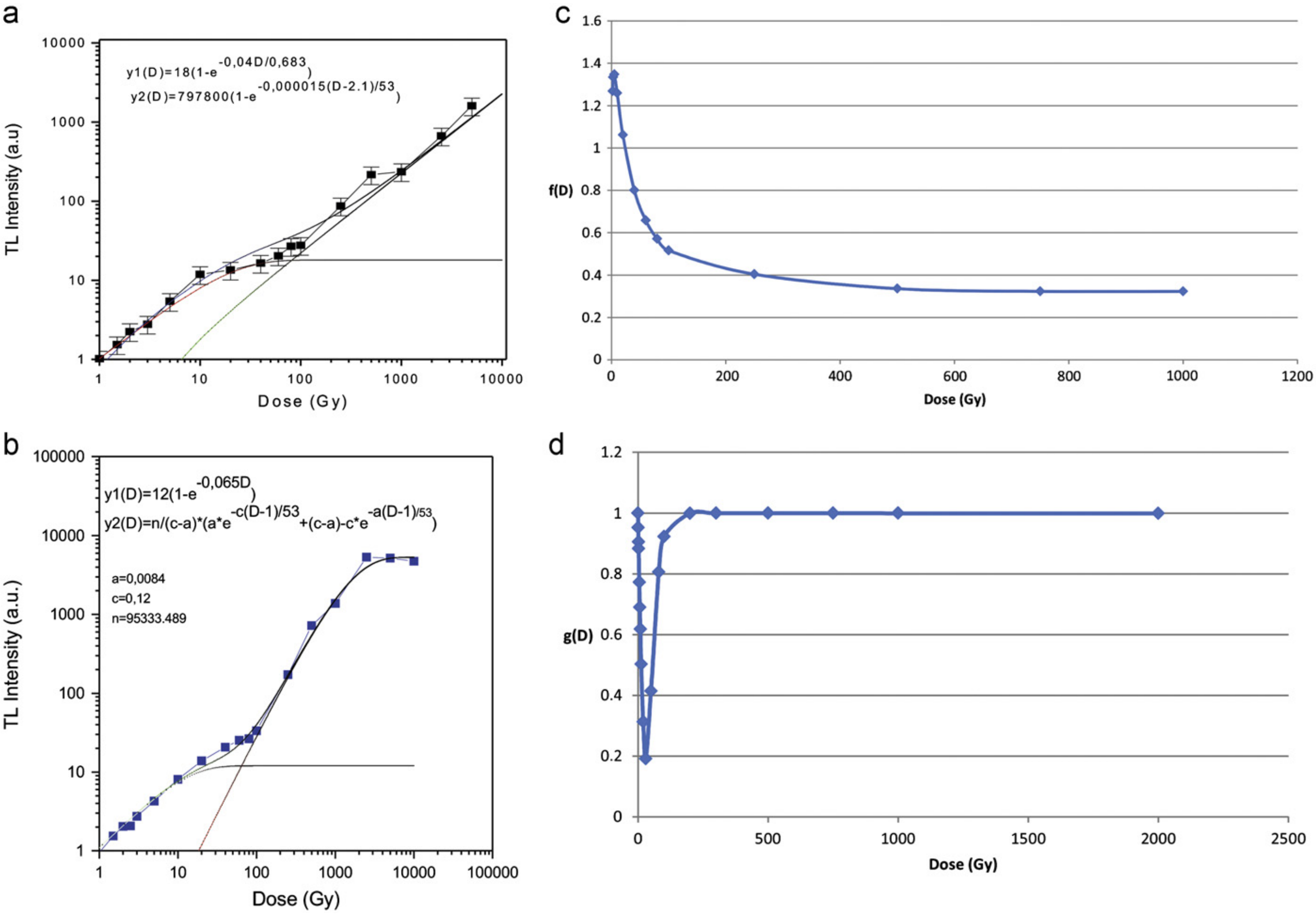

Fig. 5. (a) TL intensity as a function of gamma dose blue quartz. (peaks 325 °C). (b) TL intensity as a function of gamma dose blue quartz. (peaks 220 °C). (c) Curve of *f*(*D*) in blue quartz. (peak 325 °C). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

in the sulphurous and red quartz no high temperature peaks are observed.

As mentioned previously on the possible mechanism for production of peaks at 220, 325 and 375 °C, based on [20] model for creation of E'_1- centers involving oxygen-vacancies created thermodynamically and by radiation, the production of TL peaks requires hole recombination center. Toyoda and Ikeya have shown in addition that Ti and Al substitute Si in $[SiO_4]$ tetrahedron giving rise to the Ti-center and Al-center in the following way. We take the case of the aluminum, which is trivalent, and so after replacing tetravalent Si^{4+} in $[SiO_4]^0$ the charge disequilibrium is compensated by usually present monovalent M^+ ion (Li^+, Na^+, K^+, H^+).

$$[SiO4]^0 + Al^{3+} + M^+ \rightarrow [AlO_4/M^+]^0$$

The irradiation at room temperature removes M^+ leaving the so called aluminum-center, $[AlO_4/h]^0$. A similar reaction creates titanium-center, $[TiO_4/h]^0$. According to [20] the Ti-center decreases after 150 °C heating being extinguished at about 250 °C. For Al-center this happens between 200 and 450 °C. In both cases, electrons liberated from $[VO/2e^-]$ and $[VO/e^-]$-centers recombine with holes in Ti and Al centers, giving rise to 220, 325 and 375 °C TL peaks. VO stands for oxygen-vacancy. There is also formation of $[H_3O_4/h]$-center since OH and H_2O are always present.

Since Ti is found in all seven varieties, the appearance of 220 °C peak can be accounted for, however the fact that Al is also found in all of them, it is difficult to explain why the 325–375 °C peaks practically do not exist (or are very small) in some of the varieties of quartz.

In the TL emission spectra, the 340–360 nm band observed in the present quartz samples should be the same to 370–380 nm band found in the literature and 440–470 nm band is the same as the 470 nm band. According to McKeever [11], the 470 nm band is a photon emitted during the recombination of an electron with the hole in $[AlO_4/h]^-$ center, whereas, the 380 nm band is a photon emitted in a recombination with the $[H_3O_4/h]^-$ center. The TL emission spectra shown in Fig. 3 are TL emission spectra of the alpha, the artificial, the pink and the green quartz; Fig. 4 show TL emission spectra of the blue, the black, the milky and the sulphurous quartz. The 470 nm emission in the pink quartz is not small, nevertheless it is still four times weaker than the 340–360 nm emission. These results indicate that the recombination involving $[H_3O_4/h]$ center is predominant and the aluminiun center has a relatively smaller role in the TL emission. In all other quartz samples, both bands are much weaker than the bands observed in the alpha, in the pink and in the green quartz. In other words, the role of aluminiun centre may not play a major role in TL emissions. Is it possible the $[H_3O_4/h]$ center be found abundantly in quartz samples? It is very likely as it is well known that H_2O and OH are always present in any varieties of quartz. They are found in the well known channels along *c*-axis in quartz.

4. Supralinearity

Found in the years 1960, in LiF:Mg, the supralinear behavior of ionic crystals have been subject of a large number of models to explain.

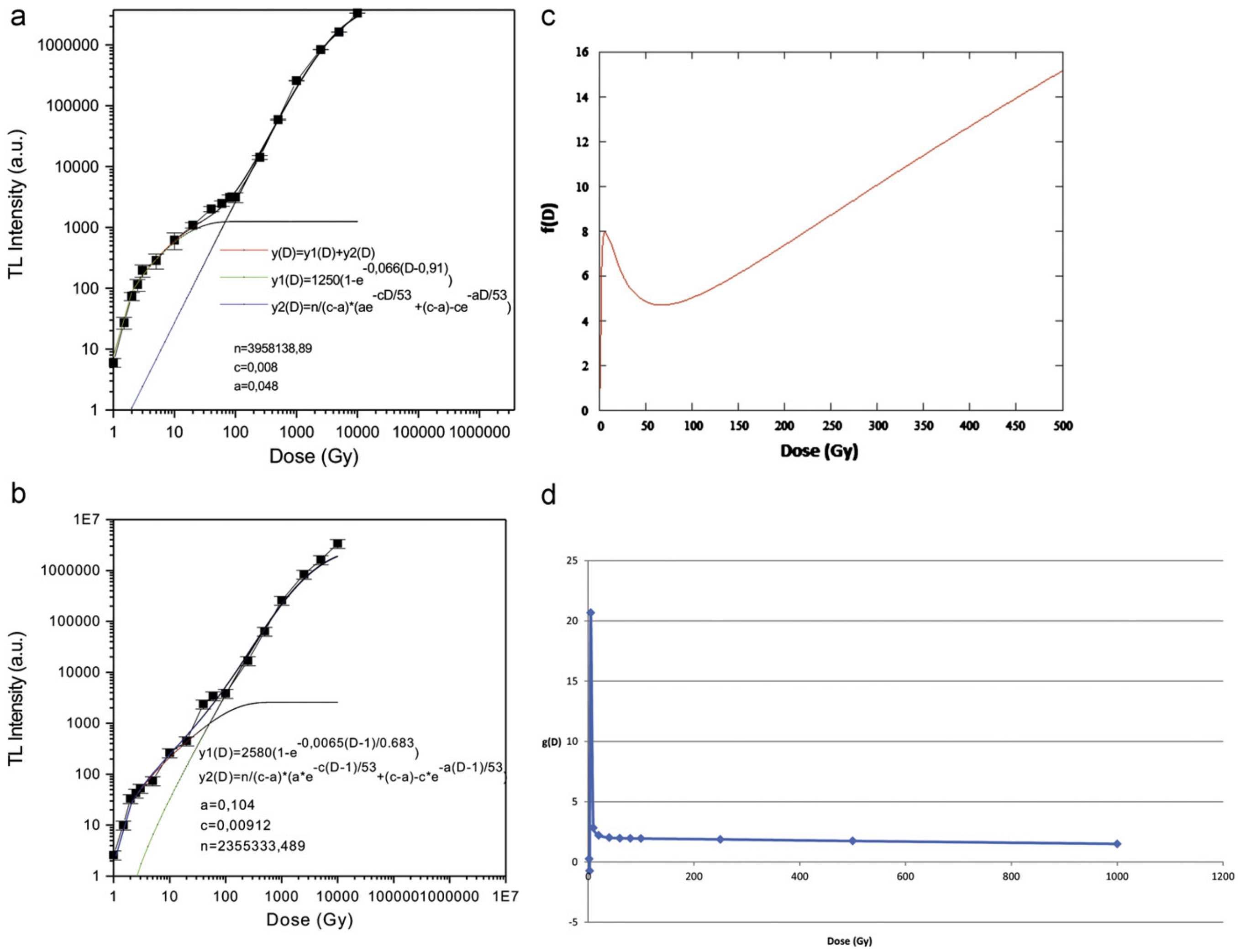

Fig. 6. (a) TL intensity as a function of gamma dose sulphurous quartz. (peak 325 °C). (b) TL intensity as a function of gamma dose sulphurous quartz. (peak 220 °C). (c) Curve of $f(D)$ in sulphurous quartz. (peak 325 °C). (d) Curve of $g(D)$ in sulphurous quartz. (peak 325 °C).

The characteristic behavior found in LiF (TLD-100) and in many other ionic crystals is that of an initial linear dependence on the dose, followed by more than linear growth and saturation. This more than linear behavior was called supralinearity. Horowitz [23], Rudra [18] and Mische and McKeever [25] quantified above property by defining the dimensionless quantity:

$$f(D)=\left[\frac{S(D)/D}{S(D_1)/D_1}\right] \tag{1}$$

where $S(D)$ is the TL signal as function of dose D, and D_1 is a dose in the linear range. We have linearity for $f(D)=1$, supralinearity for $f(D)>1$ and sublinearity for $f(D)<1$.

Chen and McKeever [24] suggested to use the term supralinearity for the increase in the derivative of $S(D)$, they introduced the supralinearity index:

$$g(D)=\left[\frac{DS(D)}{S'D}\right]+1 \tag{2}$$

where $g(D)>1$ indicate the supralinear region, $g(D)<1$, sublinear region and $g=1$, usually means, the linearity in a range or at point.Here, the linearity–supralinearity–sublinearity is discussed in term of $f(D)$.

Customarily, the TL vs dose curve is plotted in log–log scale. The TL growth behavior as function of radiation dose in the alpha quartz has been investigated by [21]. The 325 °C peak, that is important in archaeological and geological dating, presented a typical linear–supralinear–sublinear behavior. Based on model proposed by [12] for F-center formation in KCl,

Watanabe et al. [21] proposed that in quartz, the TL vs. dose curve is formed by two stages. The first one, called linear region, is due to already existing oxygen vacancies, which capture two electrons during an irradiation giving rise to a complex which we designate as $[\mathrm{OV}/2e^-]$, which OV means oxygen vacancy. The second stage is due to creation of additional oxygen vacancies by irradiation and formation of new $[\mathrm{OV}/2e^-]^-$centers. In both stages the formation of $[\mathrm{OV}/2e^-]^-$ centers is dose dependent and it was shown that, in the first stage, the TL intensity $y_1(D)$ is given by:

$$y_1(D)=y_0[1-\exp(-bD)] \tag{3}$$

where b is the rate of creation of TL traps per unit dose, and y_0 is the maximum OV centers existent at room temperature.

It is noted that in Eq. (3) has a linear behavior for low doses for $bD\leq 0.1$. In the blue quartz (220 and 325 °C peaks) and in the pink quartz (375 °C peak), the TL versus dose curve below 10 Gy is fitted by Eq. (3) and it is linear below 10 Gy. In the sulphurous and the green quartz the TL versus dose curve obeys a modified Eq. (3), namely:

$$y_1(D)=y_0[1-\exp(-b(D-D_0)] \tag{3a}$$

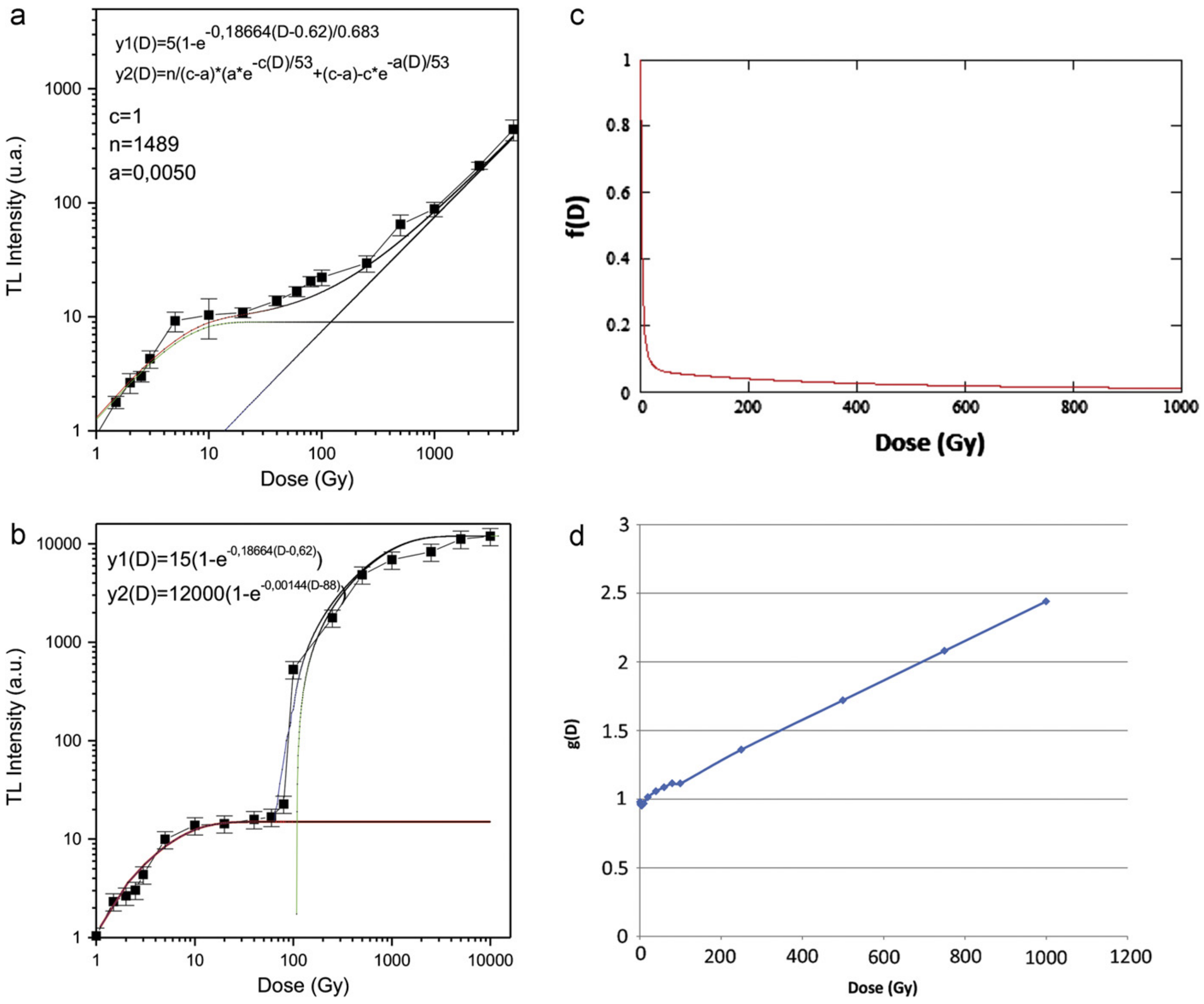

Fig. 7. (a) TL intensity as a function of gamma dose green quartz. (peak 325 °C). (b) TL intensity as a function of gamma dose green quartz. (peak 175 °C). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

where D_0 is dose value for which $y_1=0$. What is the physical meaning of D_0 was not found as yet.

In the second stage, the TL versus dose is given by:

$$y(D)=\frac{N}{(C-a)}\{a.\exp(-C.D)+C-a-C.\exp(-a.D)\}. \tag{4}$$

where C is the rate for generation of vacancies by radiation, a is the probability per unit dose to form TL trap centers by capturing electron and N is the total still available oxygen ions for generation of oxygen-vacancies. The whole TL versus dose curve is given by sum of $y_1(D)$ and $y_2(D)$, which is expected to give linearity, supralinearity followed by saturation.

5. TL versus dose response behavior of eight varieties of quartz here investigated

The model introduced by [21] explained reasonably well the TL versus dose curves for the alpha quartz at 220 and 325 °C peaks by applying Eqs. (3) and (4). The same pattern was used in other varieties of quartz described and can be seen in Figs. (5)–(9).

Fig. 5(a) and (b) shows TL versus dose response of the blue quartz, respectively for 325 and 220 °C peaks; and Fig. 6(a) and (b), for the sulphurous quartz; Fig. 7(a) and (b), for the green quartz; Fig. 8(a) and (b), for the pink quartz, and Fig. 9(a) and (b), for the black quartz for 325 °C peak and for 220 °C peak, respectively. These results can be divided into four categories:

(a) Those that are described by $y_1(D)+y_2(D)$: the alpha, the blue (220 °C peak) and the pink quartz (325 °C peak);
(b) Those that obey to $y_1(D)$ plus $y_1'(D)$ of the same form $y_1(D)$: the blue (325 °C peak) and the green quartz (220 °C peak);
(c) Those that obey to modified $y_1(D)=I_s[1-\exp(-\alpha(D-D_0))]$ but $y_2(D)$ is of Eq. (4): the sulphurous and the pink quartz (220 °C peak);
(d) Those with three stages of growth: the black and the alpha quartz;

Table 2 shows estimated values of parameters a and C in Eq. (4).

For those in category (a) the TL versus dose behavior can be described by $y_1(D)+y_2(D)$. As already mentioned, the alpha quartz requires a model for third stage of TL growth as function of dose.

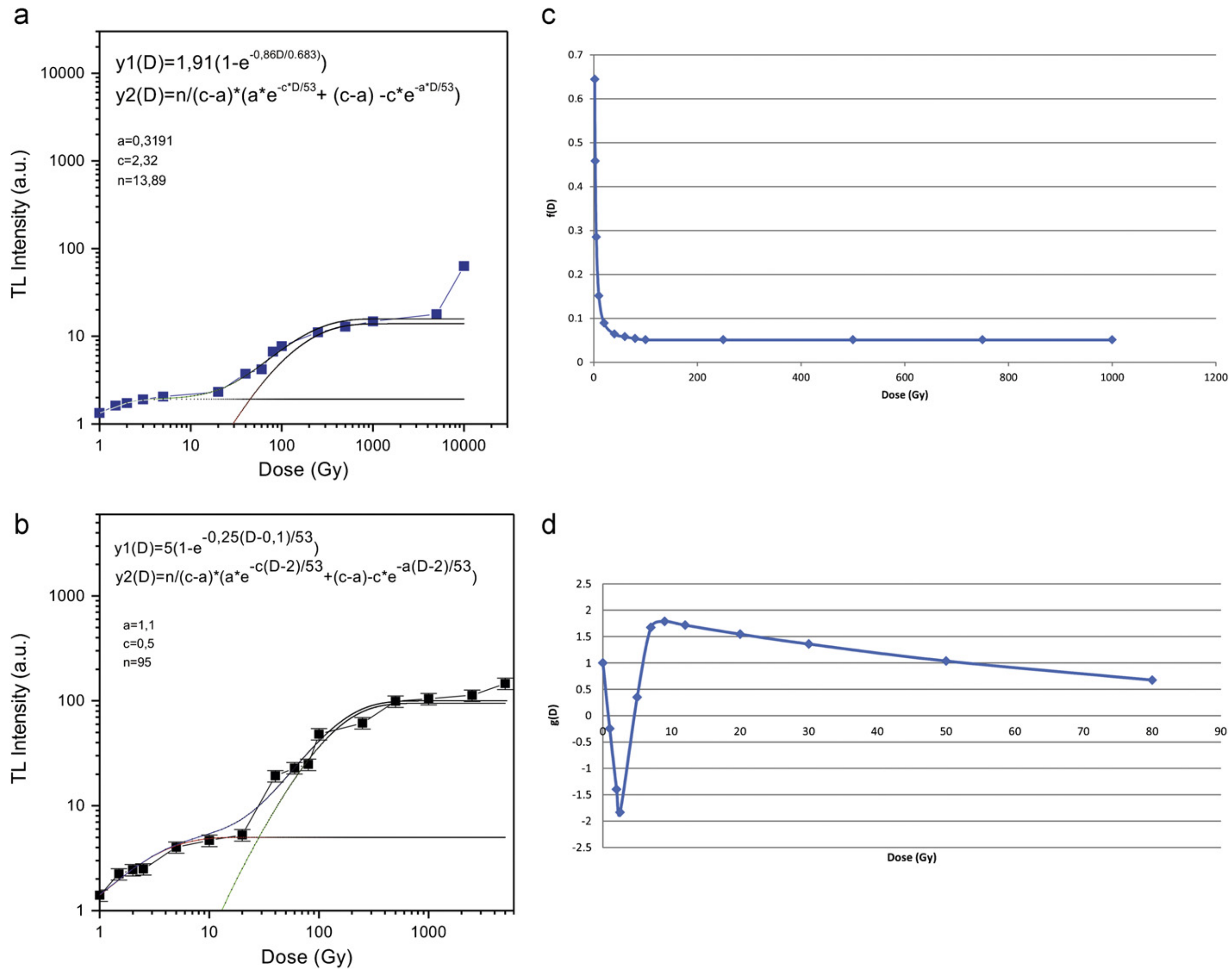

Fig. 8. (a) TL intensity as a function of gamma dose pink quartz. (peak 375 °C). (b) TL intensity as a function of gamma dose pink quartz. (peak 220 °C). (c) Curve of $f(D)$ in pink quartz. (peak 375 °C). (d) Curve of $g(D)$ in pink quartz. (peak 375 °C). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Table 2
Estimated values of parameters a and C for Eq. (4).

Samples	Alpha	Alpha	Blue	Sulph.	Sulph.	Pink	Pink	Green
Parameters	(220 °C)	(325 °C)	(220 °C)	(220 °C)	(325°)	(220 °C)	(375°)	(325°)
a	0.81	0.014	0.0084	0.104	0.48	1.1	0.2191	0.0050
C	0.015	0.00624	0.00012	0.00912	0.0048	0.5	2.32	1.0

The 325 °C peak in the blue quartz and the 175 °C peak in the green quartz have the second stage described by $y_2''(D)=I_s''[1-\exp(\alpha'(D-D_0'))]$. The 325 °C peak in the blue quartz does not show supralinearity, however the 220 °C peak in the green quartz presents a supralinearity up to about 500 Gy and then sublinearity and finally saturation.

For both 220 and 325 °C peaks in the sulphurous quartz and the 220 °C peak in the pink quartz, the $y_2'(D)$ equation replaces $y_1(D)$ in the first stage. The supralinearity above 1000 Gy is observed in the sulphurous quartz, whereas the 220 °C peak in the pink quartz no supralinearity is observed, and the saturation sets in around 300 Gy. The black quartz for 200 and 325 °C peaks, the TL versus dose growth curves occur in three stages as shown in Fig. 9(a) and (b).

Previously, it was mentioned that the sub and supralinearity regions can be expressed by Eqs. (1) and (2). By applying Eqs. (3) and (4) in Eqs. (1) and (2), we can evaluate the supralinearity index $g(D)$ as a function of dose in Eq. (2). Figs. 5–8(c) and (d) show this application in all the varieties of quartz at 325 °C peak.

It is noted, in Fig. 9(c) and (d), that for $D \leq 2.4$ Gy, the TL response is linear; and for $D=2.5$ Gy, the TL response abruptly becomes sublinear until $D=12$ Gy; then it becomes supralinear, having its maximum around 80 Gy and decreasing in the region near 3.000 Gy.

Fig. 9(e) shows the curve of $g(D)$ in the interval from 1.0 to 33,000 Gy. Note that the TL response is sublinear up to 10 Gy, becoming supralinear between 20 and 40 Gy, and above 40 Gy, it becomes sublinear again.

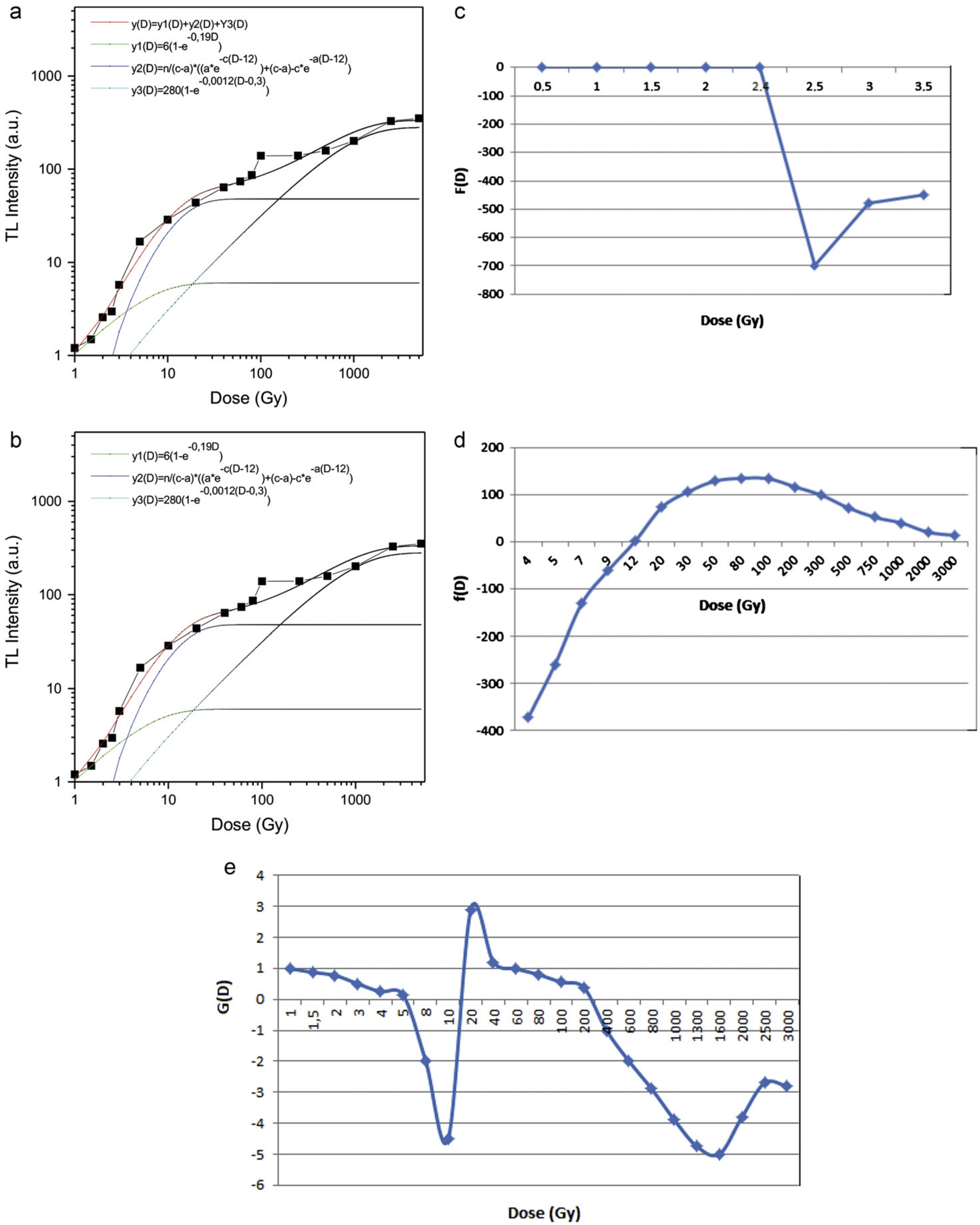

Fig. 9. (a) TL intensity as a function of gamma dose black quartz. (peak 325 °C). (b) TL intensity as a function of gamma dose black quartz. (peak 245 °C). (c) Curve *f*(*D*) for $D \leq 4$ Gy in the black quartz. (peak 325 °C). (d) Curve *f*(*D*) for $D > 4$ Gy in the black quartz. (peak 325 °C). (e) Curve of *g* (*D*) in the range of 1.0 to 33,000 Gy in black quartz.

Note that although $f(D)$ and $g(D)$ show linear, supra or sublinear behavior, as algebraical curves they are different from each other.

6. Discussion and conclusions

Seven varieties of natural quartz have been investigated concerning their thermoluminescence properties and have been compared with the alpha quartz. As impurities of Al_2O_3 is contained in all the varieties with concentration varying from 0.07% in the sulphurous quartz up to 0.82% in the green quartz; 20 ppm of Na_2O is found in all the varieties, except in the green quartz; 0.241 w% of TiO_2 is found in the blue quartz and in all others, varying from 10 ppm as found in the milky quartz up to 80 ppm in the case of the black quartz. These impurities plus H_2O and OH participate in the emission of TL light.

The alpha and the blue quartz have similar glow curves with TL peaks at 220 °C and around 350 °C (325 and 375 °C); all the others presented glow curves different in some way compared to the alpha quartz glow curve. The sulphurous and the red quartz presented only one peak around 180 °C.

The behavior of TL response to the radiation is even more complex than the glow curves of the different varieties of quartz. The linear-supralinear–sublinear-saturation characteristics explained by [21] model, is not observed in others varieties of quartz except for 220 °C TL peak in the blue and the pink quartz. Quite varied behaviors are observed in others varieties of quartz and the cause is not known yet.

The black quartz presented TL versus dose result (curve) composed of three stages. The alpha quartz also presents three stage behavior concerning TL versus dose response with an interesting property: The 325 °C peak did not saturate at 10,000 Gy gamma dose.

Whereas the alpha and the pink quartz have an intense TL emission band around 350 nm and weak band around 470 nm; the milky quartz presented an intense band around 470 nm, all the others have weaker TL emission band. The 350 nm band is attributed to *H*-center $[H_3O_4/h]$, and the 470 nm band is due to Al-center $[AlO_4/h]$.

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