IJPAS Vol.03 Issue-04, (April, 2016) ISSN: 2394-5710
International Journal in Physical & Applied Sciences (Impact Factor- 3.960)

THE PRE-DOSE EFFECT IN NATURAL ROCKS WITH HIGH-LEVEL OF PHOSPHATE

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Abstract-In the present investigation we have investigated sensitization of a natural phosphate sample, as a function of pre-gamma dose and post-irradiation annealing treatment at a test dose of 1Gy. The sensitization stabilized for pre-doses higher than 1kGy.Apre-gamma dose of 1kGy followed by 450^{o}C post-irradiation annealing treatment yielded the maximum sensitization factor of 22.The sensitization of the thermal annealing treatment was estimated to be 2.33.The position and shape of the thermo luminescence glow curves in unsensitized and sensitized samples were different.

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1.**Introduction**

Natural minerals play an important and emerging role in modern science and technology. Most of these exhibit thermoluminescence (TL) characteristics and facilitates to understand various damage and safety processes during irradiation accidents[1]. Thermal and radiation treatments have a profound influence on the thermoluminescence properties of the most of the TL sensitive phosphors. The pre-dose feature has been effectively utilized in various applications; (i) archaeological dating, (ii) retrospective dosimetry, (iii) authenticity testing, (iv) firing temperature measurements etc [2-4].

By the pre-dose, one mean the change of sensitivity, namely the response to a given test dose, by a heavier irradiation, followed by a thermal activation. Charitids et al. [5] studied the dependence of the TL dose response on the pre-dose delivered to the sample.Chen et al [6] and Charitids et al. [7] showed the pre-dose conditions, also the authors showed the effect of high pre-dose on superlinearity. Since many minerals such as phosphate, phosphate and quartz invaluable in the industry because of their combination of physical and thermal properties. The pre-dose effect first study on quartz [5-12] and then expanded to other materials [13-15]. The pre-dosed TL signal has been useful in case of applications involving low dose equivalent dose measurements, few tens of mGy to a few Gy. This happens due to early onset of saturation in case of R-canters, reservoir centers. So the growth curve, sensitization with pre-dose, initiates linearly with an increase in pre-dose up to a few grays and saturates thereafter. The simulation results also showed that the pre-dose technique can reproduce the paleodose received by the sample accurately in the low dose region of 0–2 Gy[16]. The nature of the thermal activation curve (TAC), essentially, reflects the distribution in activation energy of the reservoir traps, R. As the activation temperature is increased more and more holes get transferred to the L canters, luminescence centers. The thermal treatment involved in the pre-dose treatment has been reported to introduce pure thermal sensitization also apart from it charge transferring role [17]. The sensitization process gets reversed with UV stimulation but is restored back with subsequent thermal activation. This phenomenon is termed as UV reversal [18].

Phosphors owe their practical importance to their property of absorbing incident energy and converting it into visible radiations [19]. The study of absorption and emission characteristics of a phosphor enables the understanding of electronic energy levels in the crystal and, in turn, the design of new phosphors of practical relevance .TLD badges, which carry specially tuned phosphormaterial, are widely used in personnel as well asenvironmental radiation monitoring. Though a largenumber of organic and inorganic materials exhibit thermoluminescence,only a small fraction possesses all the ideal characteristics of a good TLD phosphor [20, 21]. Phosphates and halo-phosphates, used for luminescence applications, were also explored for their suitability as TLD materials [22, 23]. Schipper et al [24] studied trapping of electrons in the X-ray storage phosphor, $Ba_3 (PO_4)_2$: Eu, La. Seshagiri et al [25] studied the thermally stimulated luminescence (TSL) and electron paramagnetic resonance (EPR) properties of calcium chlorophosphates. The possible mechanism of TSL glow is proposed by correlating the spectral characteristics and the thermal stability of the radical ions. TL of mixed rare earth phosphate powder was studied by Michel et al [26], who observed that the TL emission spectra of the phosphor mainly composed of characteristic line transition of rare earth ions. Calcium phosphate crystals also are candidate materials for TLD applications [27].

The purpose of the present paper is to study the TL sensitization characteristics of natural phosphate. The fundamental features of the sensitization procedure can be divided into three parts:

Part 1.Study of the activation temperature.

Part2. Study of the activation dose.

Part3. Study of the annealing time.

2. Experimental

phosphate in powder form (particle size 100 µm) was provided by Astronomy and Geophysics Research Institute, King Abdulaziz City for Science and Technology (KACST) in Riyadh and originated from Hazm Al Jalamid area, in the northern region of Saudi Arabia. Its chemical formula is (P_2O_3).

The sample was washed with 1N hydrochloric acid; after that distilled water was used to remove the HCl and inorganic impurities. The sample was then allowed to dry .Magnetic particles were removed using Franz Magnetic Separator. After washing, drying and removal of magnetic particles, the phosphate sample was encapsulated in plastic capsules and kept in dark untilmeasurements were achieved.

TL measurements were monitored using a Harshaw 3500 TLD reader. Light pulses were detected by the photomultiplier tube provided with a narrow band blue filter plus Schoot BG39 glass filters of blue- violet transmittance band. A linear heating rate of $5^{o}Cs^{-1}$was chosen; heating the sample from room temperature up to $400^{o}C$. The incandescent background was measured then subtracted from the data. To minimize the statistical error, five aliquots each of 7 mg were used for each measurement. All irradiations and measurements were performed in King Saud University, Saudi Arabia.

3. Results and discussion

3.1. Natural TL

The phosphate showed a strong natural TL signal during the first cycle of heating to 400°C (Fig. 1). With a heating rate of 5°Cs^{-1}, the glow curve showed one peak at 275°C.

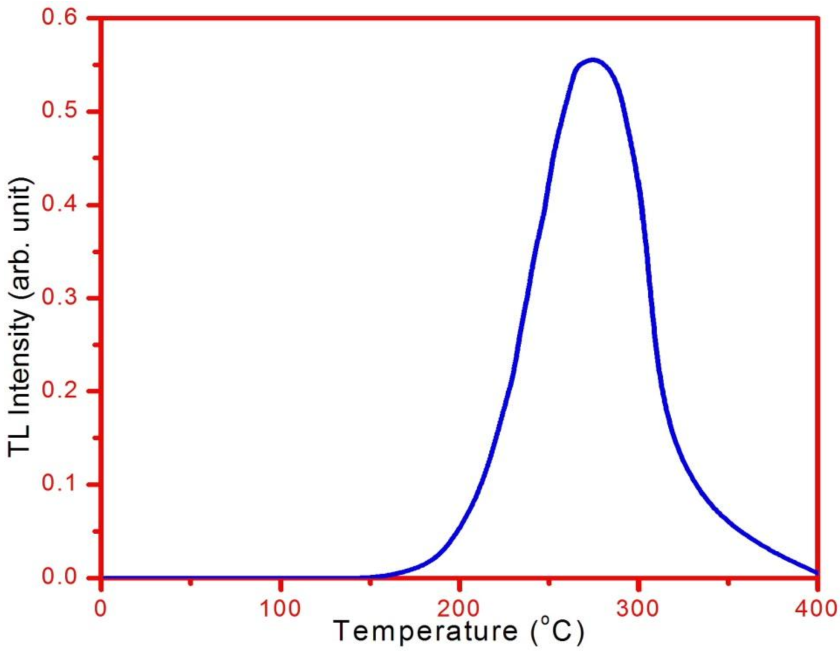

Fig. 1. The TL glow curve of natural phosphate.

3.2. Sensitization

The sensitization of TL, is the change of sensitivity of a sample to a given test dose, resulting from its exposure to a prior irradiation followed by a thermal annealing. The effect was first observed by Cameron [28].

The investigated material was first fired at 1000°C for 9 h to ensure that the deep traps are emptied, and then Fleming's sensitization technique [29] was achieved. The fundamental features of the sensitization procedure can be divided into three steps:

i) Study of the activation temperature, ii)-Study of the activation dose, iii) - Study of the annealing time.

i)The activation temperature was studied in the following way:

a) Fired aliquots (at 1000°C for 9 h) ideally of equal weight were given a test dose of 0.01Gy to measure S_o.

b) These aliquots were then given a random pre-dose 500Gy.

c) The previous aliquots were then heated at a certain temperature beginning at 250 ^{o}C and ending by 700 ^{o}C.

d) After each heating, when cooled a test dose (1Gy) was given to measure S. The sensitivity factor $S/S_o$was then calculated at different temperatures in the above mentioned range (Fig.2a). For the investigated material, 450 ^{o}C became the purification temperature for pre-dose thermal activation.

ii).The activation dose was studied in the following way:-

a) A series of aliquots of fired phosphate, were given a single pre-dose of gamma radiation in the range from 0.25kGy to 1.5kGy.

b) The aliquots were then heated to the activation temperature 450 ^{o}C.

c)When cooled, a test dose (1Gy) is applied to the previous heated aliquots which give sensitivity S and the ratio S/S_oshows a plateau at the pre-dose of 1kGy. Fig. 2b shows the variations of the ratio $S/S_o$with the pre-dose.

iii)The effect of annealing time on sensitivity is studied as follow: Powder aliquots of fired phosphate were given a dose of 1kGy and heated at 450°C to different time intervals. The aliquots were then removed and air-cooled. A test dose of1Gy was then applied, after which they were measured for TL sensitivity. The effect of this treatment is to reduce the possibility of R center saturation and maximize the sensitizing effect of the γ-ray dose [18]. The results indicate that TL enhancement is time dependent and reaches its maximum after a heating period of 15min (Fig.2c).

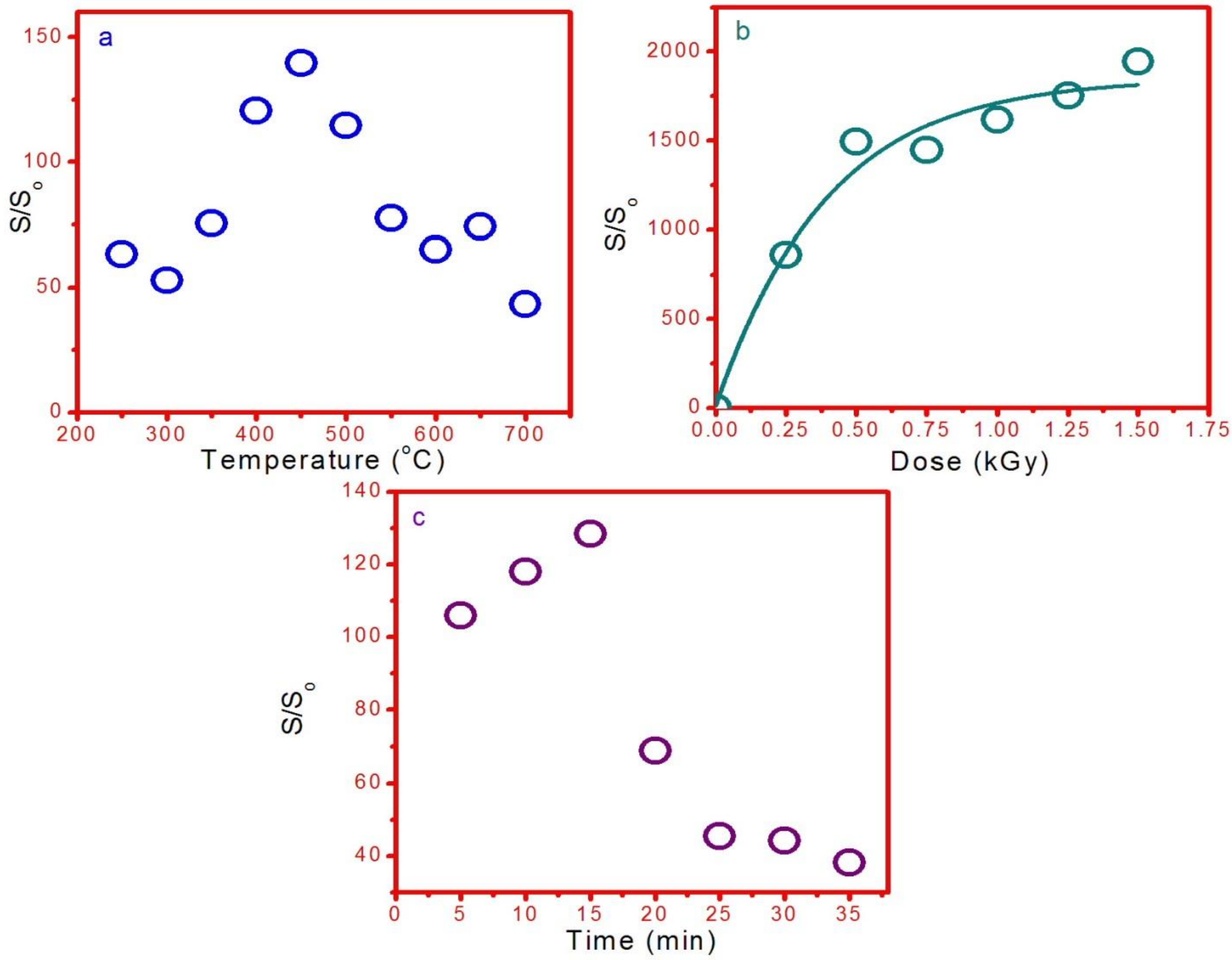

Fig. 2. Variation of S/S_o as a function of: (a) activation temperature, (b) activation dose, and (c) annealing time. For each of them the test dose was 1Gy.

Fig. 3 showsthe variations of the glow curve with the pre-dose, it must be noticed that all the glow curves have themain peak at 150°C and other high temperature peak at 275°C.

The previous steps can be summarized as follows: A pre-dose of 1kGy, to fired phosphate followed by thermal activation at 450°C for a period of 15min, and cooling in air is the optimized condition for TL sensitivity enhancement. This procedure increases the sensitivity ratio $S/S_o$by values depend upon the test dose i.e. for a test dose of 1Gy S/S_o~3.6 and for a test dose of 1000Gy S/S_o = 56.2 .This result can be explained by Chen [30]. He argued that the measured response to a test-dose is a monotonically increasing function of the concentration of trapped holes in center following the large excitation plus thermal activation.

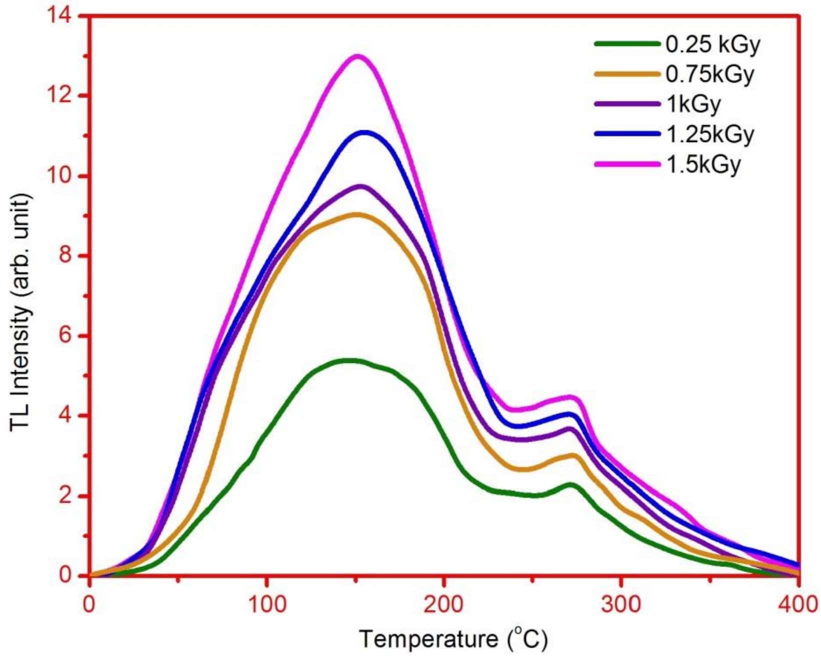

Fig. 3: The variations of the glow curve with the pre-dose.

3.3 Glow curves

The glow curves of unsensitized and sensitized phosphate (Figs. 4& 5) can be explained by using the models [18,30] as follows: unsensitized phosphate annealed at 350°C (Fig.4) and exposed to different doses (0.1 -1000) Gy, phosphate exposed to low doses can be considered to have one trapping state; at 150°C and one recombination center. The consideration discussed here follows the filling of the active trap 150°C in the absence of the competitors, and assumes that the resulting TL intensity is proportional to this filling. Qualitatively, at low doses the filling is linear(Fig.6a) .This is observed in glow curves of small applied doses .At a certain dose 10Gy, the competing trap 275°Cappeared,hence lesselectrons are made available to the active trap 150°C. This causes a lower filling of this trap .The transition region from linear to thesublinear would, however, appear science this transition obviously occurs continuously. This describes thelinear –sublinear dose response curve of unsensitizedphosphate.

TL glow curves of sensitized phosphate arepresented in Fig. 5. It is clearly indicate that sensitizedphosphate have 4 peaks at 75°C,110°C, 150°C and 275°C.The growth of these peaks are nearly equivalent. It is observed that the TL sensitivityofphosphate is drastically increased after sensitization. It is also noticedthat, though the TL intensity of unsensitized and sensitizedphosphateincreases with increase in gamma dose there is no shift in peaktemperature. The constancy of peak temperature with increase ingamma dose confirms that the peaks are of first-order kinetics.

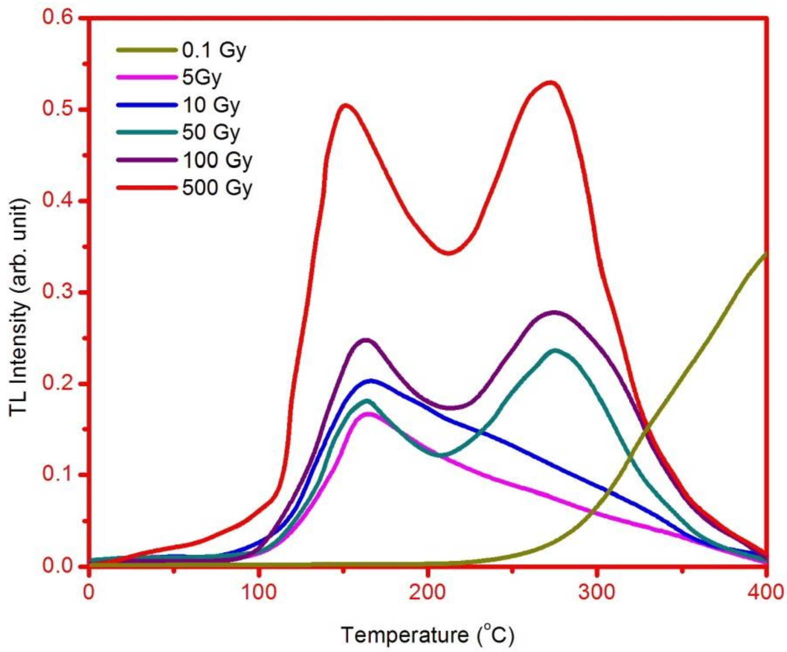

Fig.4 .Variation of glow curves of unsensitized phosphate with the γ- dose radiation.

3.4. TL Response

TL response curves of unsensitized and sensitized phosphate exposedto different doses of gamma rays are given in Fig. 6. The area under glow curves has been used for measuring the TL intensities. On exposing the unsensitized phosphate to doses ranging from 0.1Gy to 1000Gy, TL dose response of unsensitised (Fig. 5a) involved two behaviors: linear (Y = 0.3585X + 4.808) in the range 0.01 – 10 Gy thensublinear (Y= $2.4897X^{0.459}$)in the range 50–1000 Gy. Where Y corresponds to the TL intensity and X is the given dose in Gy. The sensitized mica (Fig.5b) was found to display two linearbehavior: (Y = 2.9428X + 14.289) in the range 0.01 – 10 Gy and (Y = 3.3244X + 39.736) in the range 50–1000 Gy.

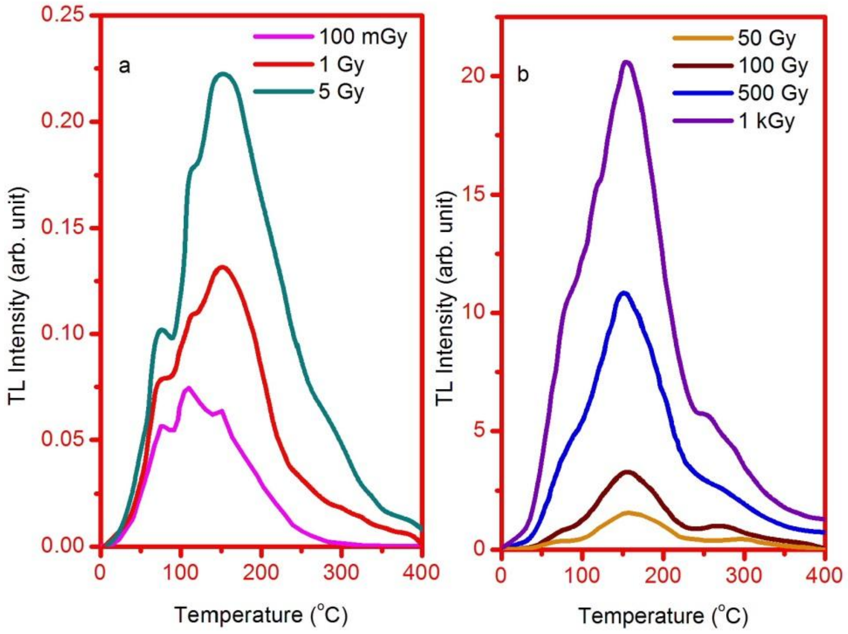

Fig.5. Variation of glow curves of sensitized phosphate with the ?-radiation.

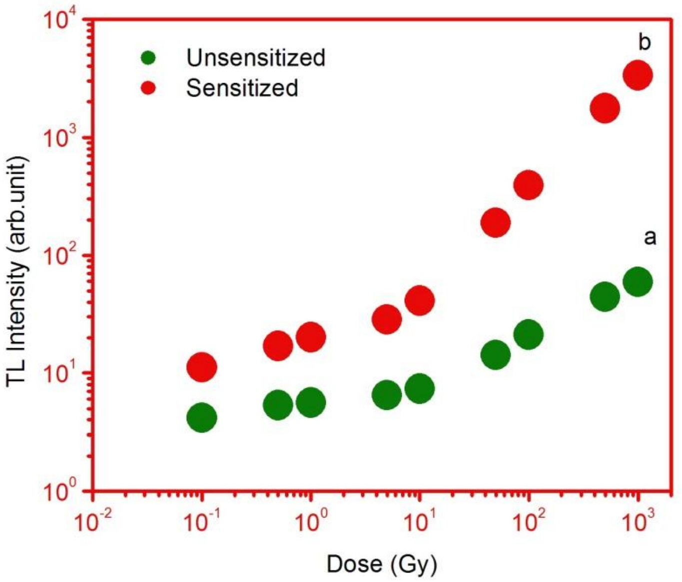

Fig.6. Growth curves of phosphate as a function of gamma dose. (a)-unsensitized sample, (b)-sensitized sample.

3.5. *Storage effects*

TL fading was performed as follows: sensitizedphosphate powder was divided into two parts: the first part was irradiated with a test dose of 1Gy and then stored for varying lengths of time in dark at room temperature. Successive measurements were monitored over a period of 14days. The other part was used as control aliquots by immediate storage to avoid variations from instrumental drift.

Thereafter, they were irradiated with dose identical to that before and read out immediately after irradiation. The stable TL signal for the aliquots exposed to 1Gy was stored in dark was found to be~20%after a delay period of 14days (Fig.7).

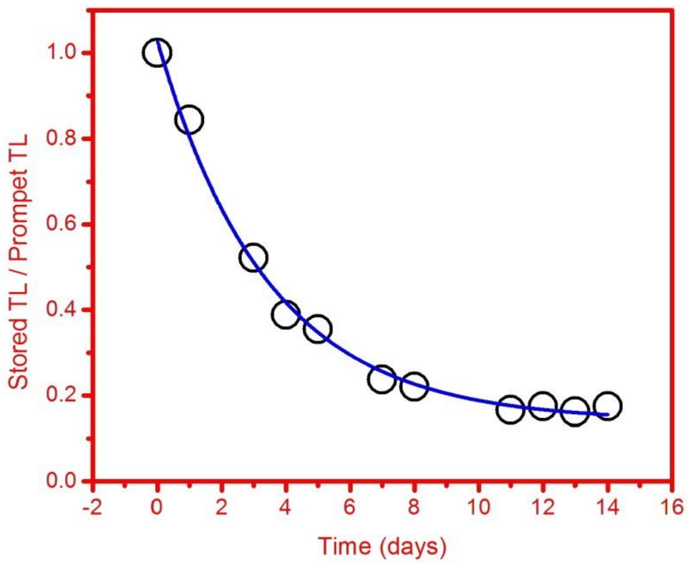

Fig.7 TL fading of sensitized phosphate as a function of time at room temperature.

4. Conclusion

In this work, the sensitization characteristics of phosphatehave been studied. It is found that the sensitization is greatlydependent on pre-dose, temperature and the duration of theheat-treatment. A pre-dose of 1 kGy followed by heat treatmentat 450^{o} C for 15 min yields the maximum sensitization factor forphosphate sample. The behavior of TL glow curves priorand after sensitization has been investigated. TL response curvesof unsensitized and sensitized phosphate showed different behaviorwhich has been explained. The comparisonof TL glow curves and response curves of unsensitized and sensitizedphosphate indicates that sensitization has greatly enhancedthe TL sensitivity of phosphate.Fading study showed that the stable TL signal for the aliquots exposed to 1Gy stored in dark was found to be 20%after a delay period of 14 days.Post irradiation annealing should be studied to reduce the fading, which will be helpful in radiationdosimetry and other material science technology.

Acknowledgment

The authors are indebted to the Deanship of Scientific Research at Princess Nora University, for funding this research project. We extend our appreciation to colleagues in King Saud University in Riyadh, Kingdom of Saudi Arabia for providing the necessary facilities.

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