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Research Article

Tuff Rock as a New Thermoluminescent Material for Gamma Dosimetry

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Abstract

Background and Objective: Radiation dosimetry related to nuclear accidents or terrorist events suffers from disadvantages such as the time required for sample preparation and variation in the sensitivity of the samples. The objectives of this study were to investigate the thermoluminescence (TL) properties of natural tuff and to evaluate its use for gamma radiation dose measurements in retrospective dosimetry. **Materials and Methods:** Tuff is lithified volcanic ash and is produced by explosive volcanic eruptions. A sample was ground into powder and treated with HCl, H₂O₂, sodium oxalate and alcohol. TL measurements were performed using Harshaw TL equipment. Gamma irradiation was administered with a ⁶⁰Co source. **Results:** The as-received sample shows one main TL peak, while radiation and heat treatment induced 3 others. The response to gamma irradiation is linear for small doses and sub-linear for high doses. Batch homogeneity was calculated and found to meet the IEC recommendations. Storage measurements showed that fading stopped after 7 days. **Conclusion:** Indications are that this material is a potential candidate for dose reconstruction in retrospective, dosimetry and industrial applications under certain restricted conditions.

Key words: Thermoluminescence, tuff, gamma, response, homogeneity, fading

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Competing Interest: The authors have declared that no competing interest exists.

Data Availability: All relevant data are within the paper and its supporting information files.

INTRODUCTION

The risk of unexpected nuclear accidents or violent terror attacks necessitates methods and processes that are appropriate and applicable in such emergencies. Luminescence techniques have been applied to a wide range of radiological studies. Retrospective dosimetry involves reconstruction of doses received, from weeks to years after exposure. Because many individual dosimetric techniques cannot be used multiple times after exposure due to the fading of the corresponding radiation-induced signals, the peculiar process of retrospective dosimetry has been developed. This is the reconstruction of spatial patterns of radiation-doses humans could have received during environmental exposure via assessment of actual doses to buildings and structures. This is accompanied by modelling to take account of population movements and behavior¹. One technique for this is the thermoluminescence (TL) method, which is commonly applied for the detection of ionizing radiation for environmental personal, medical and reactor dosimetry purposes². Many objects in the environment can be used as natural dosimeters, if they have suitable TL and optically stimulated luminescent (OSL) properties. Retrospective methods were used on bricks and tiles for reassessment of atomic bomb radiation dosimetry in Hiroshima and Nagasaki³. More recently, a broad variety of results from retrospective dosimetry problems were published in which materials within modern electronic and personal items (telephone chip-cards, electronic components of portable electronic devices such as cell phones, pagers and digital watches) were considered for dose reconstruction using the OSL technique⁴⁻⁶.

The TL of porcelain was investigated in order to develop better protocols for retrospective dosimetry⁷. While some glass samples showed promising results, the inherent variability of an amorphous substance such as glass means that the suitability of each sample must be determined on a case-by-case basis. TL signals may also be light sensitive, as seen by the OSL responses of Pyrex and other glasses⁸.

The possible use of natural barite for radiation dosimetry and geochronology was explored using the TL technique⁹. Salt appears to be a suitable material for retrospective dosimetry under favorable circumstances¹⁰. Kinetic analysis of barite from Australia found the lifetimes of the TL peaks to be sufficient for application to retrospective dosimetry¹¹.

The potential application of TL-OSL to accident dosimetry was investigated using Japanese surface soil¹². Additional studies of barite in dust were needed to more accurately assess the minimum detectable dose and to confirm the

general applicability of the method for accidental situations¹³. The basic TL and OSL properties of schist were studied to evaluate its potential use in archaeological dating and retrospective dosimetry¹⁴.

The present study evaluates the new TL material natural tuff as a natural dosimeter. The TL characteristics considered necessary for its critical evaluation as a retrospective dosimeter were analyzed. For that, authors used 3 kinds of measurements: TL glow curves (laboratory induced as well as natural), TL-gamma dose response curves and 2 weeks monitoring of storage effects.

MATERIALS AND METHODS

This study was carried out from October, 2016 through June, 2017 at King Saud University, Kingdom of Saudi Arabia (KSA) and King Abdul-Aziz City for Science and Technology (KACST) in Riyadh.

Material: The natural tuff rock sample was from the town of Afif (KSA) and was provided, prepared and chemically treated at KACST. Tuffs are dominated by ash grains with lapilli or blocks or bomb clasts. Most tuffs form due to the accumulation of ash some distance from a volcanic vent by direct air fall from the volcanic plume.

Methods

Preparation of the sample

Grinding: The sample was first ground and sieved to the desired diameter (100 μ m).

Remove magnetic grains: A Franz magnetic separator was used to remove magnetic grains.

Chemical treatment: To get rid of carbonates and fine grains, the sample was treated with 1 N HCl and 30% H₂O₂ for 2 h and then deflocculated using 0.01 N sodium oxalate. The grains were then settled in alcohol using the Fattahi and Stokes method¹⁵. The alcohol was evaporated by heating at 45°C, leaving the samples on aluminum discs.

Thermal treatment: For thermal treatment, each powder sample was placed in a quartz bowl inside an electronic oven and annealing was performed at different temperatures (100-700°C) for 30 min. At the end of the annealing interval, the sample was allowed to cool rapidly to room temperature and was kept in the dark until the TL measurements were performed. After exposing a sample to a gamma (γ) radiation dose of 5 Gy for 30 min, TL glow curve readouts were taken.

TL measurements: The TL measurements were performed in the range from room temperature 20-22 to 400°C (Harshaw-3500 TL, USA). All TL glow curves were recorded using a blue filter, of which transmission dropped to 1% for wavelengths longer than 500 nm and an infrared-rejecting filter. All TL measurements were performed with a linear heating rate of 5°C sec⁻¹ and all the data represent the average of at least 5 runs performed with different aliquots of the tuff powder.

Gamma irradiation: The doses required to measure the growth curve were administered using a ⁶⁰Co source, with doses ranging from 100 mGy to 1000 Gy.

Statistical test: Data presentation and analysis were performed using the programs origin 2016 and TLanal.

RESULTS AND DISCUSSION

Glow curves: At sufficiently high temperatures, TL signals of the as-received sample started to show the doses accumulated in the tuff, without introducing laboratory irradiation. The background signal of the sample and the reader were then recorded for a second readout. The glow curve of the as-received sample did not change shape, but did show minute intensity variations. When tuff was subjected to heating from room temperature to 400°C, at a linear heating rate of 5°C sec⁻¹, the glow curves showed one peak at 310°C (Fig. 1). In the natural sample, low-temperature trap centers were found to be empty, because the low-temperature filled traps fade fast with time a fact observable even if the sample is kept at room temperature. The TL signals in the as received sample are typically attributed, in part, to self-irradiation, in addition to irradiation from the surrounding environment. This result is close to the result from previous work on volcanic ash from India, which had a glow peak at ~320°C and which is very close to the present result¹⁶. However, this is far from another result that showed a single trap at ~150°C¹⁷.

Thermal treatment: Aliquots annealed at different temperatures (100-700°C) were exposed to γ -dose irradiation of 5 Gy for 30 min, then TL glow-curve readouts were taken. At this stage, significant changes in the glow-curve shape were clearly observable (Fig. 2).

Two peaks were observed (at 175 and 310°C) when tuff was annealed at 100°C. When the annealing temperature was

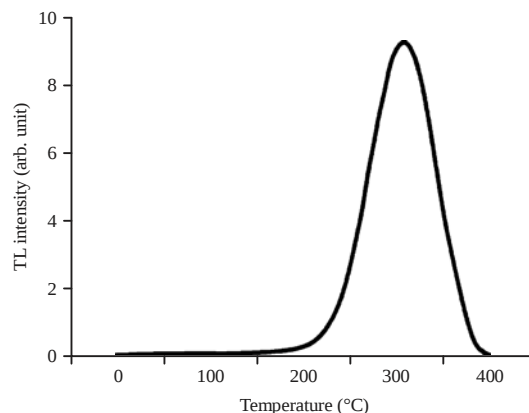


Fig. 1: Glow curve of the received tuff recorded at a heating rate of 5°C sec⁻¹

increased from 200-700°C the first peak position was the same while the second peak had shifted to 250°C. As can be seen in Fig. 3, the sensitivity of the tuff annealed at 300-400°C for 30 min was minimal, meaning that the acceptor traps had emptied.

The results from this study indicated that the best annealing temperature was 400°C (error 5%). Band theory implies that these peaks are attributable to traps found in the crystal forbidden band gap. The thermal treatments have the ability to alter the defect topography, producing different glow curves in both natural (Fig. 1) and laboratory-treated aliquots (Fig. 2). The latter show that the early filling of the traps at the temperature peak of 175°C was because they had already been emptied, hence, they were first in line to fill.

The curves in Fig. 2 show that there were two activation temperatures. The first was 300°C, at which the peak at 175°C was sensitized and the second was 700°C, at which the peak at 250°C was sensitized. Chen *et al.*¹⁸ illustrated this observation when they presented their 'competitor during heating model. In the material under investigation here, we can explain, in a straightforward manner, the increase in sensitivity following heating to high temperatures. Once competitors have been removed, or made ineffective, sensitivity will eventually increase due to the fact that the charges released from traps will travel to the luminescence sites.

This result contradicts results from previous work with volcanic ash¹⁶ in which the optimal preheating temperature of 325°C was used. The other type¹⁷ from the Indian Ocean used preheat temperatures >120°C, which is also far from the current results.

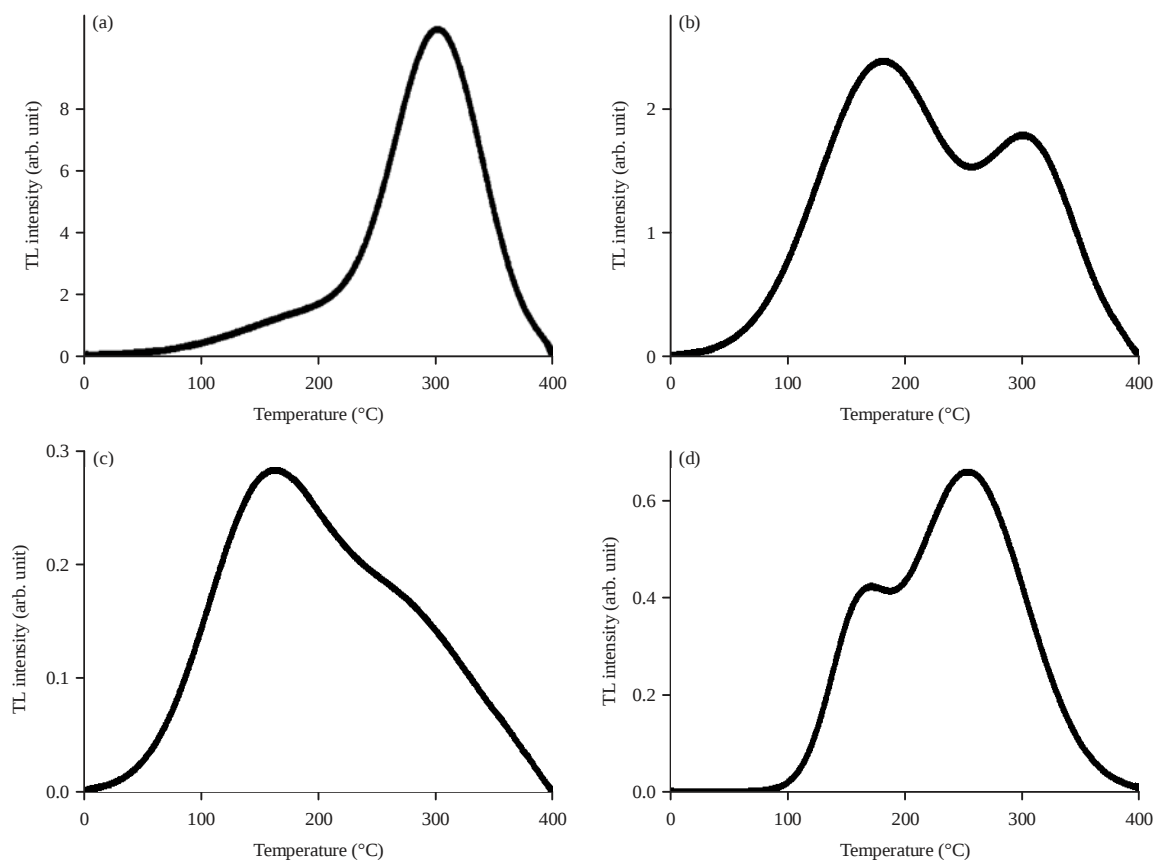


Fig. 2(a-d): TL glow curves of the blue-band of tuff pre-annealed at different temperature (a) 100°C, (b) 300°C, (c) 500°C and (d) 700°C,

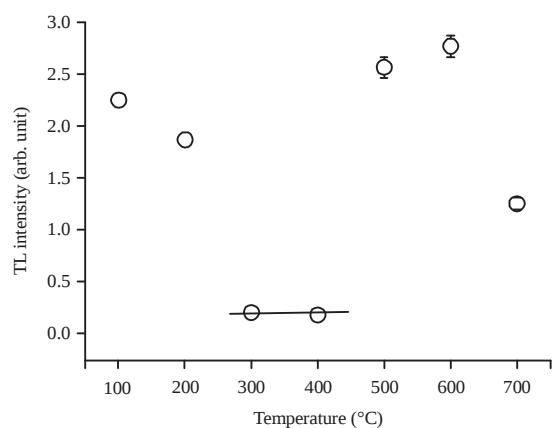


Fig. 3: Variation of the γ response of tuff with the pre-heating temperature (error bars are SD)

Effect of γ -irradiation: Tuff annealed at 400°C was γ -irradiated with doses in the range from 100 mGy to 1000 Gy. Four peaks were obtained in the laboratory-induced glow curves. One appeared at 65°C at the small dose of 100 mGy, then shifted towards higher temperature (135°C) as the dose

increased after exposure of 1000 Gy. The second peak appeared at 110°C at a dose of 3 Gy. The peak shifted a little (to 170°C) with increase in the exposure dose (1000 Gy). Third and fourth peaks appeared at 310 and 360°C at the dose of 3 Gy, but did not change position even after exposure of 1000 Gy. Kristianpoller *et al.*¹⁹ described this as 'strong competitor re trapping'. Band theory implies that these peaks are attributable to radiation-induced defects in the crystal forbidden band gap. The difference between the heat-induced TL peaks and others induced by radiation (Fig. 2, 4) are the three peaks at 65, 110 and 360°C, which can be considered indicators of radiation-induced defects. The peaks at 310 and 360°C were saturated at doses up to 50 Gy, after which no response to laboratory gamma ray irradiations was observed.

TL response vs. dose: Figure 5 shows the TL dose response of tuff in the blue band, in the dose range 100 mGy to 1000 Gy. The TL growth curve in the blue band of tuff shows a two-phase process. The first phase, ranging from 100 mGy to 5 Gy, exhibited linear behavior in the form ($Y = 316 + 46.4D$,

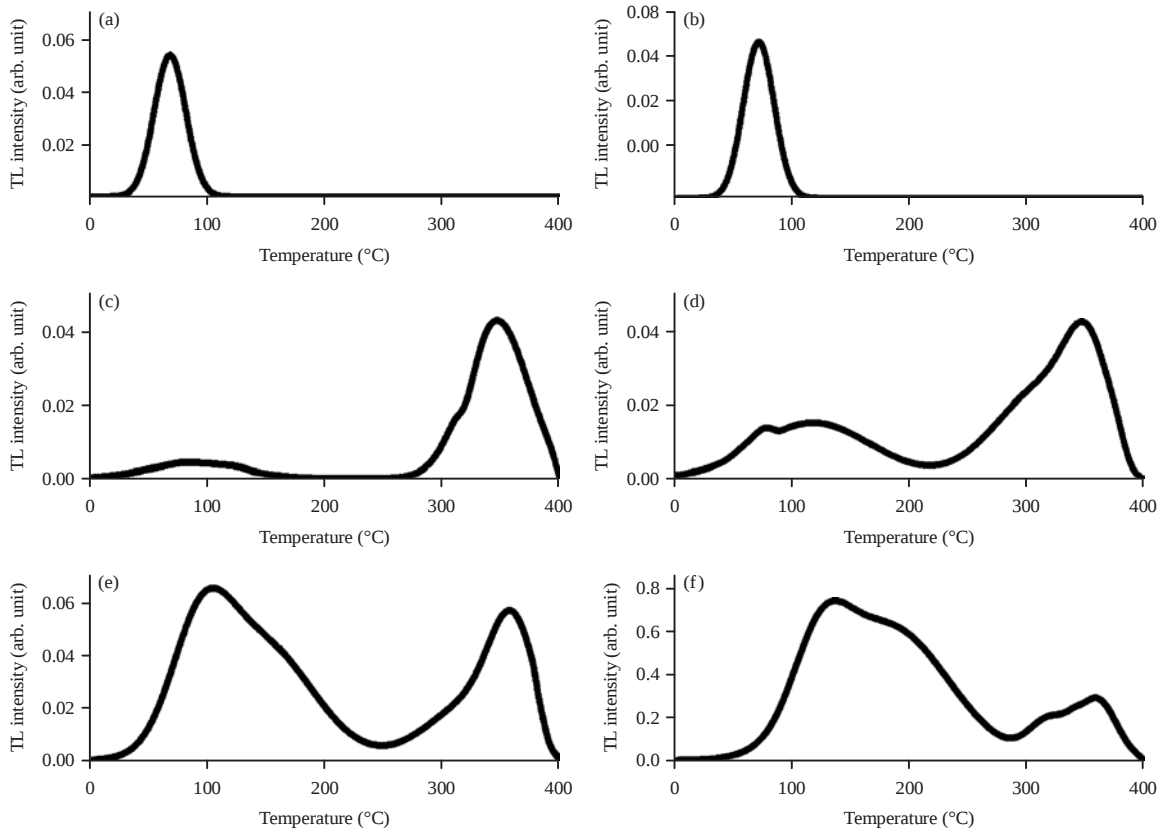


Fig. 4(a-f): Variations of the TL glow curves of tuff with the irradiation γ dose, (a) 100 mGy, (b) 1500 mGy, (c) 3 mGy, (d) 10 mGy, (e) 50 Gy and (f) 1000 Gy

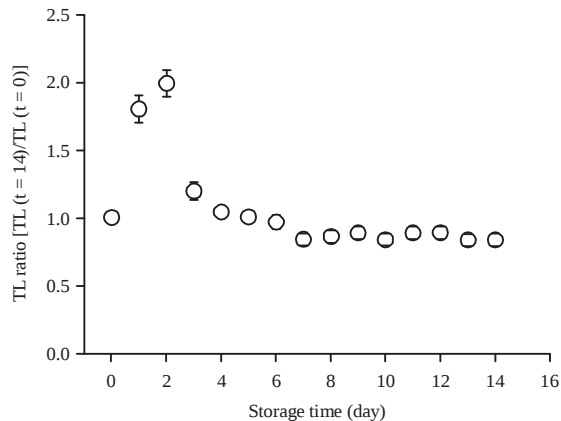


Fig. 5: Variability TL intensity with increment of gamma-radiation dose in tuff (error bars are within data)

$R^2 = 0.999$). In the second phase, ranging upto 1000 Gy, the TL emission showed a sublinear relation ($y = 115.6D^{0.65}$, $R^2 = 0.976$) with the irradiation dose, where Y corresponds to the TL intensity per g, D is the irradiation dose in Gy and R^2 is the correlation coefficient. Although the dose response is

nonlinear for most thermoluminescent materials, these results demonstrate that a number of nonlinear effects result from variation in the irradiation type and the range of doses²⁰.

Volcanic ash showed a minimum detectable dose of 5 Gy and onset of saturation dose of 330 Gy¹⁶. Another sample exposed to doses in the range 66-2112 Gy exhibited an exponential response¹⁷.

Batch homogeneity: It is normal to find discrepancies in TL responses coming from different aliquots of a material, despite its being subjected to the same treatment. The international electrochemical commission (IEC) recommends that these discrepancies not exceed a limit 30% for different dosimeters of the same batch²¹. Fifty samples from a single batch of tuff served to verify this condition. f for tuff was calculated and found to be similar within 0.160.07, which meets the IEC limit:

$$f = \frac{TL_{\max} - TL_{\min}}{TL_{\min}} \leq 0.3$$

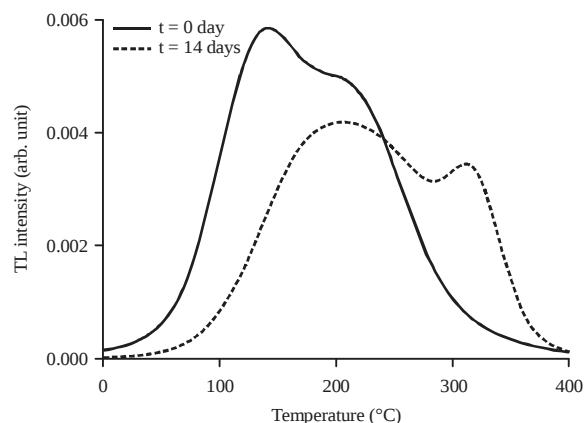


Fig. 6: TL response glow curves of tuff after different storage periods

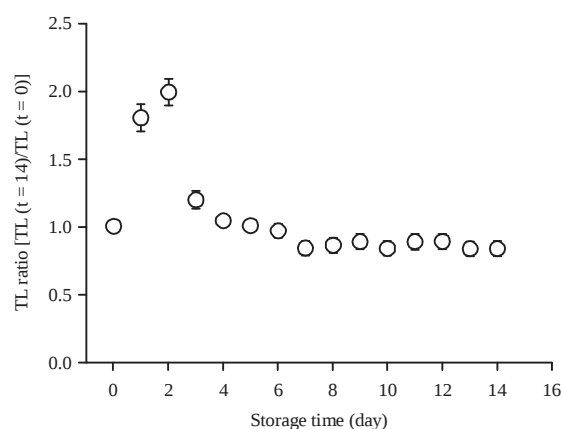


Fig. 7: Remnant TL of tuff as a function of storage time at room temperature (error bars are SD)

where, f is the batch homogeneity coefficient, TL_{max} is the max. reading of the aliquot, TL_{min} is the min. reading of another aliquot after undergoing the same treatment in all steps (annealing, irradiation and readout). To quantify and illustrate the homogeneity of the phosphor in a certain batch, it was calculated that the percentage standard deviation of the 10 TL responses from their mean, which came out to be 4.5%. The percentage standard deviation of the TL responses illustrates the homogeneity of the tuff samples.

Sensitivity: It was studied the tuff's sensitivity in comparison with an ideal dosimeter TLD-100. Numerous tuff aliquots were measured for this purpose, with the result that a relative sensitivity $R(D)$ was created and defined by Chen and McKeever²⁰:

$$R(D) = \frac{S(D)_{tuff}}{S(D)_{TLD-100}}$$

where, $R(D)$ was estimated to be around 0.3 in the dose range 100-300 mGy, 0.75 in the dose range 500-1500 mGy and 0.15 in the dose range 1-5 Gy. This result may be attributed to the higher TL light self-absorption in tuff powder than in the TLD-100 chips. This could be one reason why the sensitivity of the tuff is lower. In order to work on the phosphor's sensitivity, it might be useful to find a convenient combination of treatments though pre-exposure and/or annealing²⁰. Such combination will be the focus of forthcoming endeavors.

Stability of traps: The stability of traps is a parameter that closely reflects a trap's storage capacity. For the purpose of investigating stability, TL measurements were taken over a 14 day period at room temperature. Prior to storage, the investigated material was given an annealing treatment at 400°C for 30 min. A suitable amount of annealed tuff powder was irradiated (5 Gy) and stored in the darkness before TL reading. Another amount of the annealed sample was used as control after being stored for a period of 14 days under the same conditions, then irradiated with the same dose and read out immediately.

As author noticed in Fig. 6, there exists a shift in the glow-peak temperature with time of storage. One explanation of this could be that glow-peaks at lower temperatures, after a certain storage time, undergo faster decay than those at higher temperatures, taking into account that these at lower temperatures probably are of notably higher intensity. Figure 7 displays the relationship between the storage time and the remnant TL. A small growth in the TL was observed within the first 2 days storage time. This is thought to result from the migration and aggregation of low-temperature trapping centers to form more stable complex centers²². Fading started rapidly, but the rate decreased with increasing storage time. With time, the TL signal fell to ~84% of its original value. This time dependency springs from the idea of a potential barrier for which the probability of tunneling decreases exponentially with increasing separation between recombination and trap sites and with the random spatial trap-center distribution²³. The fading rate of the previously studied volcanic ash samples was found to be high 1.6% per decade¹⁶ and $9.6 \pm 3.5\%$ per logarithmic decade¹⁷. A summary of TL survey of the previous work on similar materials is presented in Table 1.

The contradiction in results between our investigated material and the similar materials in Table 1 can be attributed to differences in the measuring photo multiplier, filters, environment, annealing conditions, heating rates and impurities.

Table 1: A summary of the TL survey of previous work on similar materials

Sample No.	Peak position (°C)	Dose response range, fitting	Fading factor (%)	Reference
Loess	50, 290	Upto ~3000 Gy, nonlinear	3.6±1.3	Thiel <i>et al.</i> ²⁴
Feldspar	200, 250	Upto ~2000 Gy, nonlinear	Negligible	Li and Li ²⁵
Feldspar	100, 300	Upto ~1600 Gy, nonlinear	-	Jain and Ankjærgaard ²⁶
Meteorite	230, 370	-	9.5±1.1	Biswas <i>et al.</i> ²⁷
Feldspar 305, 375	-	-	3- 30	Guerin and Visocekas ²⁸
Tuff	-	Upto ~600 Gy, nonlinear	-	Lepper <i>et al.</i> ²⁹
Eruption	-	Upto ~2600 Gy, nonlinear	-	Zhao <i>et al.</i> ³⁰

CONCLUSION

The retrospective dosimetry of gamma rays in the range 0.1-5 Gy in the blue emission band of tuff indicates its promise as a phosphor. One aspect of this is that the occurrence of TL emissions occurs from ~50-400°C, after which it undergoes complete annealing. The ongoing readout procedure appears to provide resistance to the effects of different environmental factors. The TL glow curves of the tuff showed varied forms with variation in the laboratory treatment, which might be used for dose discrimination. All this comes to good use on turning attention to the possible applications of this phosphor. One example might be nuclear-accident-related dose measurement (reconstruction). In order to work on the phosphor's sensitivity, it might be possible to find a convenient combination of treatments though pre-exposure and/or annealing. This will be addressed in forth coming endeavors.

It is recommended that, in future endeavors, attention be focused on trying to split up individual peaks from the broad overall one and to conduct conclusive studies about each peak, about sensitization parameters and about the use of preheating temperatures suitable to overcoming fading effects.

SIGNIFICANCE STATEMENT

This study reveals that tuff rock can be beneficial as a new phosphor for use in retrospective dosimetry in nuclear accidents, under certain restricted conditions. This study will help researchers uncover the usefulness of volcanic materials for use in radiation dosimetry that many researchers have not yet been able to explore. This study serves as a recommendation that specialists in every country implement programs to investigate the further use of natural materials as retrospective dosimeters.

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