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Original article

Annealing Temperature Effects on the Thermoluminescence Glow Curve and Kinetic Parameters of the Natural Soil Salt Phosphor from Zuwara Sabkhas in Northwestern Libya, as Radiation Dosimetry

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Abstract
In this work, the thermoluminescence (TL) properties and kinetic parameters of soil salts that collect on the soil surface after rainfall are studied. The samples were crushed and pressed carefully to form circular disc pellets before being irradiated with a calibrated 90Sr-90Y source-β-particles emission source 1Gy. The optimum annealing condition was determined by varying the annealing temperature from room temperature (RT) up to 700oC/1h. TL responses exhibited two peaks at about 175°C and 235°C in the glow curve. By increasing the annealing temperature, we observed that the glow curve has a single sharp peak at about 225 °C and a broad shoulder at high temperature. The phase crystalline structure of the soil salt was analyzed by the powder X-ray diffraction technique. These phosphors exhibited a high sensitivity, which might prove useful for TL dosimetry in applications.
Keywords. Natural Soil Salts, Natural and Doped Salt, Effect of Annealing, TL-Glow Curve.

Introduction

A phenomenon known as thermoluminescence (TL) occurs when a specimen absorbs energy from a previous irradiation and then releases some of that energy as light when heated. If Chen and McKeever [1] record the glow curve, which shows the intensity of emitted light against temperature, they can learn more about the charge release mechanisms and recombination processes because the detected light is directly related to the energy emitted by the trapped electrons. Typically, defects and distinct impurity sites within the crystalline material indicate the varied locations where electrons are trapped. However, naturally occurring minerals that have distinct TL glow peaks are generally thought to be intriguing candidates for their possible applications as archeological dating materials [2]. The widespread and naturally occurring alkali halide NaCl salt is one of these intriguing and reasonably priced compounds. This ecologically abundant material from various sources is significant and has the potential to be used for radiological event analysis and retrospective dosimetry. Because of the energy deposited in electronic and nuclear collisions, it is well known that NaCl, like other alkali halides, is extremely sensitive to ionizing radiation. Actually, TL is a method for assessing materials of the semiconductor and insulating types; for example, McKeever [3-5]. The use of the TL technique for possible applications in retrospective dosimetric studies has been a summary of the numerous early works, attempts, and investigations that have focused on the TL of the well-known salt (NaCl). However, several reports and follow-up studies, such as the work done by, have focused on this sensitive phosphor material [6-8].
The present study focuses on determining the TL characteristics and the dosimetric properties of the commonly extracted natural NaCl salt samples collected from the Zuwara Sabkh and subjected to different annealing temperature conditions.

Experimental and Method

Sodium chloride (NaCl) is found in natural salt that is harvested from the Sabkha soils. Samples were extracted from the Zuwara area; Zuwara City lies in Northwestern Libya, as shown in (Figure 1). The Zuwara Sabkhas in Libya are known for their high salt content in the soil, which is a natural characteristic of these coastal salt flats. These soils, also known as salt flats, form in areas where evaporation exceeds precipitation, leading to the accumulation of salts.
The process of extracting salt traditionally from the present study focuses on determining the TL characteristics and the dosimetric properties of the commonly extracted natural NaCl salt samples collected from the Mediterranean Sea water and subjected to different annealing temperature conditions. Through the evaporation of seawater or salt lakes, from the soil, when it rains in the winter, the Sabkha is filled with rainwater, and the water level rises until it floods the entire Sabkha area. The rain continues to fall in these large lakes until late spring, announcing the start of the water evaporation season, and with the gradual temperature rise, leading to the formation of salt crystals, as the collection process begins in early May, and the people shovel these layers resulting from the evaporation of salt water with heavy machinery, and in the past it was done with simple agricultural tools and collected in a pyramidal shape, and then pulled out of the water swamps.

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In this study, natural soil salt (SS-NaCl) samples were used. These samples were gathered and kept in a dark environment. A fine powder was created after they were crushed. To obtain a consistent particle residue with a size of less than < 63 µm, the powder was then sieved through a mesh. The measurement samples were formed into round discs having a diameter of 5 mm and a thickness of approximately 1 mm. They each weighed 15 mg and were compressed at a pressure of 1.0 tons. After that, samples were exposed to a range of temperatures, and the effects of the annealing temperature were examined. To ensure accuracy, multiple samples, three for each batch, were generated, and repeated assessments using the same method under the same circumstances revealed comparable exposure dosage effects. By gradually letting the samples cool to room temperature (RT) before the β-irradiation procedure, the ideal annealing condition was found by adjusting the annealing temperature from RT to 700°C/1h. The efficiency of the annealing temperature in relation to the rate of stimulated charge release was the only factor used to determine the criteria of the ideal annealing condition. The probability of recombination at luminous sites was then calculated. However, it was discovered that annealing temperature at 700°C produced the greatest results for all samples.
A β-irradiation 90Sr-90Y source, which produces a dosage at a rate of 2.87 µGys^{-1}, was subsequently used to irradiate all samples at room temperature. Dosage uniformities were low and reflect a marginal uncertainty to within ± 2%, and the impacts of any dosage rate adjustments provided by the manufacturer and repeated measurements add to the reliability. Additionally, all measurements were made immediately following the administration of the dose 1Gy. A conventional Harshaw 3500 TLD reader was used to measure thermoluminescence, and the TL light curves were recorded at a steady heating rate of 2 Ks^{-1} from room temperature to 400°C. Less than 1.0°C separated the ambient temperature from 400°C within the measured range. Before and after every readout, the blackbody radiation was always measured using an empty tray, and it was deducted from all of the TL glow curves.

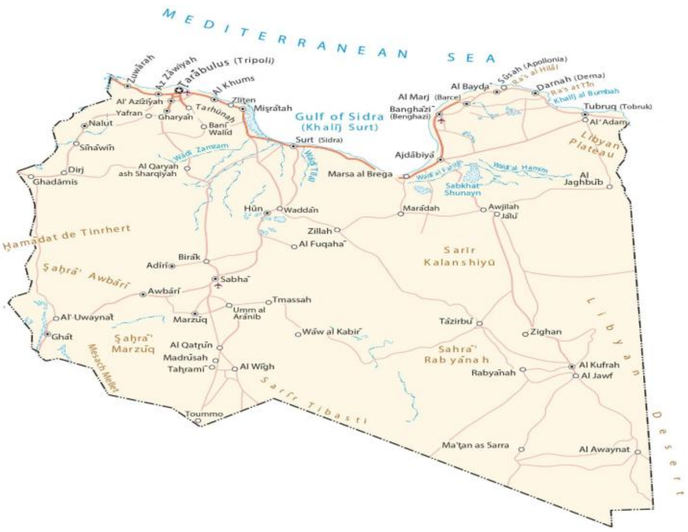

Figure 1: Location map of Sabkha site in the study area. Zuwara Sabkhas in Northwestern Libya.

Results and Discussion

Structural characterization

When one approach fails due to sensitivity, availability, or resolution limits, employing multiple techniques becomes essential to enhancing the validity of data and conclusions. To better correlate the results with the TL features of the recorded glow curves, the complementary technique of X-ray diffraction was employed in this work. Figure 2 shows typical X-ray diffraction patterns reflection planes recorded using a stepwise approach, with steps of 0.02 degrees and a counting period of 0.5 s, within the angular range of 10 to a maximum of 80. Reflection planes related to crystalline natural SS-NaCl minerals measured by XRD. The natural salt contains many mineralogical phases, according to the XRD investigations. The strong peaks described and associated with the crystalline phase (Figure 2) are matched by the distinctive peaks acquired from the analysis of SS-NaCl. Excellent agreement is found in the comparison of the NaCl structure from R070292 and R070534 in the RRUFF database. See, for example, [6,7] for the large peaks that were found between 31.52 and 45.64 degrees because of scattering from the (200) and (222) planes.

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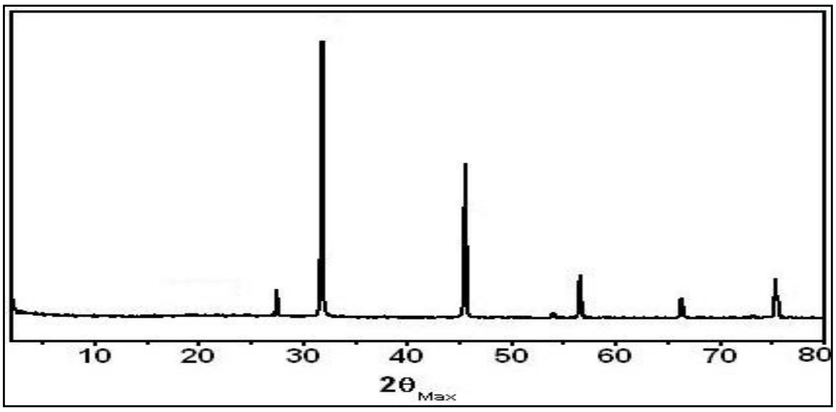

Figure 2: Typical X-ray powder diffraction (XRPD) pattern and reflection planes associated with soil salt structure.

Effect of Annealing Temperatures on TL-Glow Curve of Soil Salt

The impact of annealing temperature (Ta) on the materials' TL response up to 700°C was examined in Figure 3. (Figure 3: a,b), shows that the samples without any treatment result in TL glow curves very close to the background. After being annealed at various temperatures for one hour, each sample was exposed to a dose accumulation of one gray (Gy). It is evident from the TL glow curves that the TL response increases when the annealing temperature reaches its maximum. An in-depth analysis of Fig. 3(c-f), TL glow curves reveals identified TL traps (peaks) with differing intensities in relation to the annealing temperature. This demonstrates that the TL emitting material and TL response can be predicted with appropriate annealing temperature calibration if the TL is just dependent on the annealing temperature. The as-prepared samples result in TL light curves with the clear identification of three separate traps near 105°C, 180°C, and 220°C. At relatively annealing temperatures, near 500°C, trap satellites of varying intensities are detected near 175 and 245°C, and at 600°C, it is noticed that the 175°C trap has diminished on the expense of the 245°C trap, such that these traps and the 235°C recorded one become comparable in intensity. As the temperature is further increased, e.g., 700 °C, it is interesting to notice that the 175°C TL traps have merged with the 235°C trap to a single TL sharp trap at near 230°C with high intensity. At such high annealing temperatures, a high flux diffusion of electronic charges toward a region of high concentration of defect structure is expected. The observed high-density surface and deep defects indicate that the defect structure and charges in the disordered region are mobile within the lattice and near the region of highest defect concentration. Another possibility is that the annealing temperature induces some disturbance in the defect structure, with a clear indication of a charge transfer mechanism [6,7].

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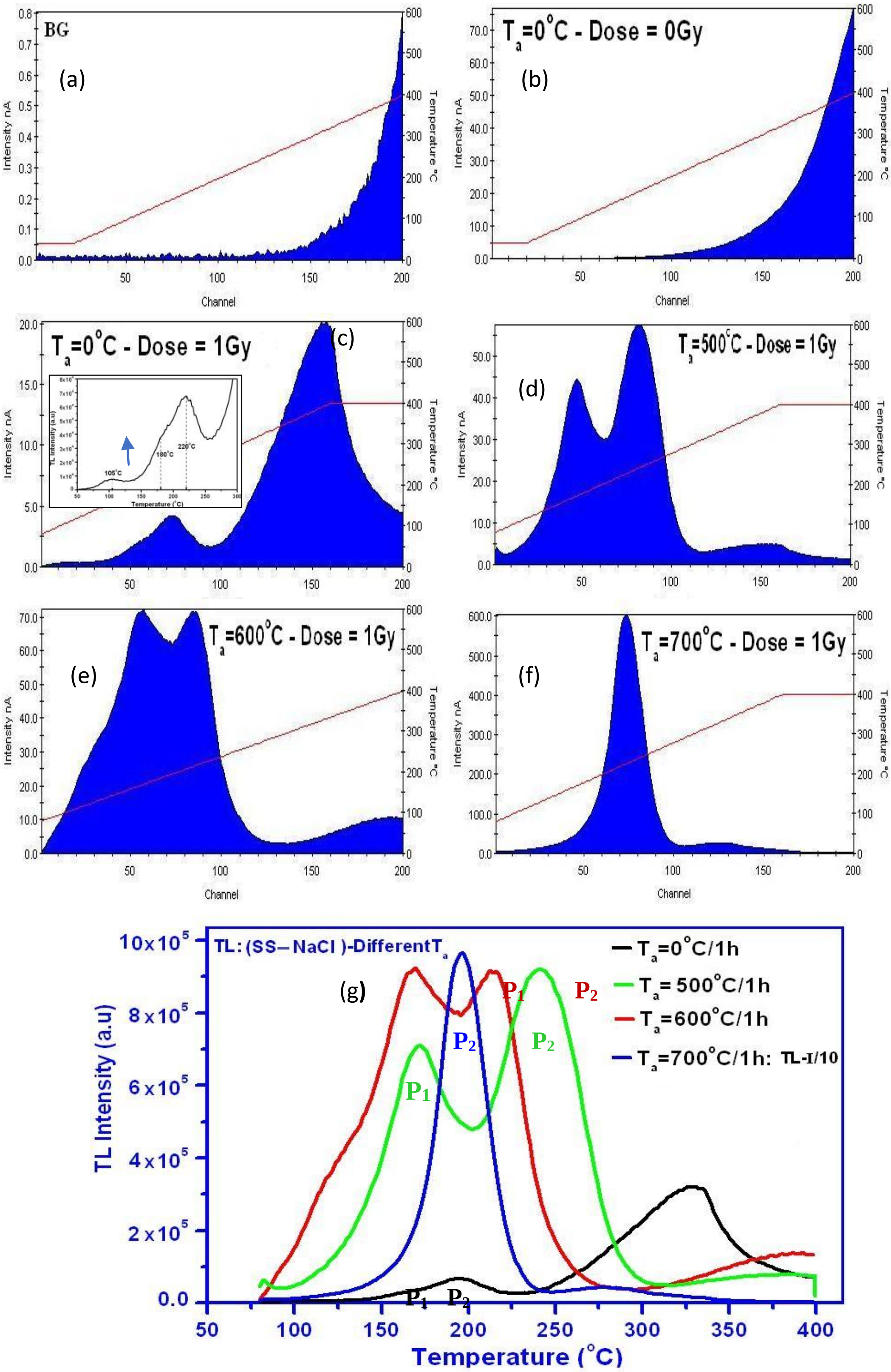

Figure 3: TL glow curves of natural soil salt (SS-NaCl) samples: as-prepared compared to blackbody radiation signal, in (b); (c) The variation in TL peaks on the imparted β-irradiation dose 1 Gy of natural SS-NaCl un-annealed; (e-f) Representative TL glow curves of β-irradiation dose to 1Gy SS-NaCl samples recorded at different annealing temperatures; (g) Typical TL glow curves of SSW as a function of the annealing temperature irradiated with 1 Gy. Measurements are recorded at a heating rate of 2 Ks-1.

Analysis of Kinetic Parameter Characterization of TL Traps: Natural Soil Salt

The computerized glow curve deconvolution (CGCD) method was used to investigate the dosimetric properties for each trap in the glow curves, and the kinetic parameters were determined. Various trap components are extracted to reveal mechanisms dominating charge transfer and recombination processes. Analysis of data, however, involves determining the trapping parameters based on available defect models and related techniques [9,10]. To understand the nature of the traps formed in natural SS-NaCl under

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annealing temperature, we have employed the analysis of Kitis et al. [11] who derived expressions for the TL glow curve GC) for first, second, and general kinetics orders using the experimentally obtained maximum trap temperature (TMax) and the maximum peak (IMax). The free kinetic parameters to be determined through nonlinear curve fitting are then the activation energy (E) and the kinetic order (b). In their analysis, the general kinetics order is generally given by [12,13]:

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

Where I(T) is the TL intensity at absolute temperature T, no is the initial concentration (cm^{-3}) of trapped charge carriers at time t = 0, and initial temperature T_0, (the irradiation temperature, RT), β is the heating rate (in Ks^{-1}); assumed linear: ($T = T_0 + \beta$ t), b is the kinetic order, E = activation energy or trap depth (in eV); $s = s'n^{(b-1)}$ o is a constant characteristic of the electron trap, called the pre-exponential frequency factor or attempt-to-escape frequency (s^{-1}). With this definition, s has the units of frequency (s^{-1}) similar to that in the first-order kinetics but depends on the applied dose rather than being a constant. (Equation 1) is solved numerically using a modified version of the program code of Chen and Kirsh [9]. It includes the second-order kinetics (b = 2) and reduces to the first-order kinetics (b = 1) as b → 1. Since b is one of the unknown parameters, we decided to use the general-order kinetics, with b may take any value between 1 and 2 [11].
In this work, fitting the experimental data of (Figure 4: a-d) with (Equation 2) using the common practice procedure of total computerized glow curve deconvolution (CGCD) to separate inherently overlapping features is an active area of interest and has become more important in view of its numerous applications, such as in dating, dosimetry, and defect studies. The method is based on solving (Equation 1) numerically using standard software, as PeakFit, Origin, or Python, and we obtained the kinetic parameters (i.e., E, TM, and b) as a function of annealing temperature. Typical result of the fitting is shown in (Figure 4: a-d) for a beta dose of 1.0Gy, revealing the detection of the additional new defect states that were absent before annealing. The well-defined nature of the glow curve at 1 Gy is manifested by the perfect fit of the theoretical curve with the experimental data. The obtained kinetic parameters and the trap positions (temperatures at the maximum) (T_M) are given in Table 1). The incandescent background was subtracted from the TL analysis data to show traps, which were overlapping with blackbody radiation.

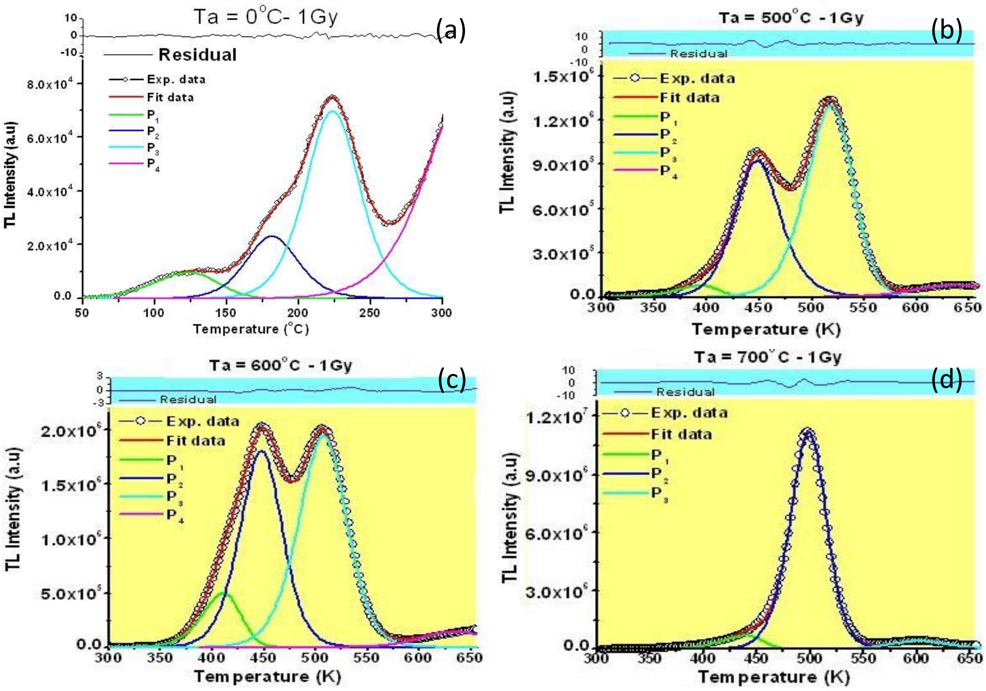

***Figure 4:** De-convolution of TL glow curves of a natural soil salt (SS-NaCl) sample annealed at: (a) as prepared* $T_a = 0℃$*; (b)* $T_a = 500℃$*;* $T_a = 600℃$*;* $T_a = 700℃$*. All samples are β-irradiated to a 1 Gy dose and measured at a constant heating rate of 2K/s.*

The process of deconvolution for natural SS-NaCl samples indicated one to two overlapping TL traps and a shoulder peak at high maximum temperature, as shown in Figure 4. The results of the kinetic parameters obtained by the GCD fitting technique are listed in Table 1. The TL glow curve of the naturally prepared SS-NaCl indicates four overlapping TL traps before annealing temperature and annealed temperatures at

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500 and 600°C/1h, respectively, as shown in (Figure 4: a-c). The TL glow curve of the natural SS-NaCl after annealing at 700°C/1h is the TL glow curve of the naturally prepared NaCl salt before annealing is best described by one main prominent TL sharp trap (peak). In n all TL glow curves, TL peaks labeled P1 and P2, see (Figure 3: g), are observed at intermediate levels of energy and deeper trap ranges: peak P1 at 180–225°C and peak P2 at 225–325°C. The relevant kinetic parameters resulting from the deconvolution process are shown in Table 1. From the deconvolution results of the glow curves fitting technique, the SS-NaCl samples annealed at 700°C which exhibit enhancement of the TL glow curve, result in one main TL peak at about ~ 226°C of mixed kinetic-order (b ~ 1.60 ± 0.01), activation energy value (E ~ 1.599 ± 0.02eV).

Table 1. Trapping parameters of TL-traps of natural soil salt (SS-NaCl) as obtained by the total GL-curve deconvolution technique.

T_a(°C)	TL-Peak	E(eV)	T_M(K)	b
0	P_1	0.625	397	1.16
	P_2	1.322	454	1.83
	P_3	1.484	497	1.74
	P_4	1.197	581	0.5
500	P_1	0.833	411	1.20
	P_2	1.046	447	1.59
	P_3	1.163	507	1.54
	P_4	1.524	633	2.00
600	P_1	0.631	394	1.00
	P_2	1.063	448	1.86
	P_3	1.210	518	1.38
	P_4	1.255	637	2.00
700	P_1	0.824	439	1.00
	P_2	1.599	499	1.60
	P_3	1.701	600	2.00

Conclusion

The dosimetric properties of natural soil salt (SS-NaCl) samples were investigated and tested for use as a radiation dosimeter for low beta irradiation doses. The thermoluminescence characteristic of natural soil salts (SS-NaCl) and the effect of annealing temperature on the SS-NaCl were studied. Annealing produces changes in the sensitivity of all SS-NaCl samples to ionizing radiation. Annealing increases the intensity of all glow peaks, which are irradiated with 1Gy. The increase in TL producing efficiency depends on the annealing temperature and irradiation. The annealing procedure reveals a change in the kinetic parameters E, Tm, and b of the glow curve. Our results showed that the annealing process probably does change the nature of the trapping centers. Heating for 1h at 700°C followed by slow cooling to ambient room temperature are the optimum conditions for TL sensitivity enhancement in the systems investigated. Based on the results obtained in this study, the SS-NaCl sample has a single dosimetric peak of the glow curve at 225 °C when heated at 700°C, indicating that soil salt can be a thermoluminescence dosimetry detector for radiation, so we recommend studying this material for adoption as a radiation dosimetry as TLD-100.

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Conflicts of Interest

The authors declare no conflicts of interest.

References

1. Chen R, McKeever SWS. Theory of thermoluminescence and related phenomena. Singapore: World Scientific; 1997. 526 p.
2. Aitken MJ. Thermoluminescence dating. London: Academic Press; 1985. 359 p.
3. McKeever SWS. Thermoluminescence of solids. Cambridge: Cambridge University Press; 1985. 563 p.
4. Spooner NA, Barnaby WS, Creighton DF, Danièle Questiaux PG, Hunter PG. Luminescence from NaCl for application to retrospective dosimetry. Radiat Meas. 2012 Sep;47(9):883-9. DOI: 10.1016/j.radmeas.2012.01.012
5. Gartia RK. Paleothermometry of NaCl as evidenced from thermoluminescence data. Nucl Instrum Methods Phys Res B. 2009 Sep;267(17):2903-7. DOI: 10.1016/j.nimb.2009.06.106

***Received**: 22-06-2025 - **Accepted**: 19-08-2025 - **Published**: 27-08-2025*

6. Khamis F, Arafah DE. Thermoluminescence Characteristics of Natural Quartz and Synthesized Silica Glass Prepared by Sol-Gel Technique. Asian J Phys Chem Sci. 2017;3(1):1-16. DOI: 10.9734/AJOPACS/2017/35542
7. Khamis F, Arafah DE. Dead Seasalt as a thermoluminescent phosphor for beta irradiation dosimetry. Appl Phys A. 2021;127:539. DOI: 10.1007/s00339-021-04463-3
8. Gartia RK. Paleothermometry of NaCl as evidenced from thermoluminescence data. Nucl Instrum Methods Phys Res B. 2009 Sep;267(17):2903-7. DOI: 10.1016/j.nimb.2009.06.106
9. Chen R, Kirsh Y. Analysis of thermally stimulated processes. Oxford: Pergamon Press; 1981. 362 p.
10. Kitis G, Gomez-Ros JM, Tuyn JWN. Thermoluminescence glow-curve deconvolution functions for first, second and general order kinetics. J Phys D Appl Phys. 1998;31:2636–41.
11. Chen R. Glow curves with general order kinetics. J Electrochem Soc. 1969;116(9):1254-7. DOI: 10.1149/1.2411628

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