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TL glow curve analysis of UV, beta and gamma induced limestone collected from Amarnath holy cave

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ARTICLE INFO

Article history:

Received 21 November 2014

Received in revised form

19 December 2014

Accepted 22 December 2014

Available online 2 January 2015

Keywords:

TL glow curve analysis

Limestone

CGCD

EDX

ABSTRACT

The paper reports thermoluminescence glow curve analysis of UV (ultraviolet), β (beta) and γ (gamma) induced limestone collected from Amarnath holy cave. The collected natural sample was characterized by X-ray diffraction (XRD) technique and crystallite size calculated by Scherer's formula. Surface morphology and particle size was calculated by transmission electron microscopy (TEM) study. Effect of annealing temperature on collected lime stone examined by TL glow curve study. The limestone was irradiated by UV radiation (254 nm source) and the TL glow curve recorded for different UV exposure time. For beta irradiation Sr^{90} source was used and it shows intense peak at 256 °C with a shoulder peak at higher temperature range. For gamma radiation Co^{60} source and TL glow curve recorded for different doses of gamma. The kinetic parameters calculation was performed for different glow curve by computerized glow curve deconvolution (CGCD) technique. The chemical composition of natural limestone was analyzed by energy dispersive X-ray spectroscopy (EDXS).

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1. Introduction

Natural samples, which are essential for dating, have TL properties that depend upon their genesis, chemical

composition, impurity content, and geological history; so a TL study is required for each sample (Nambi et al 1978). A large number of natural materials contain calcium carbonates, which explain the wide interest in using the latter to date archeological events and biotic and geological processes, for

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Peer review under responsibility of The Egyptian Society of Radiation Sciences and Applications.
<http://dx.doi.org/10.1016/j.jrras.2014.12.012>

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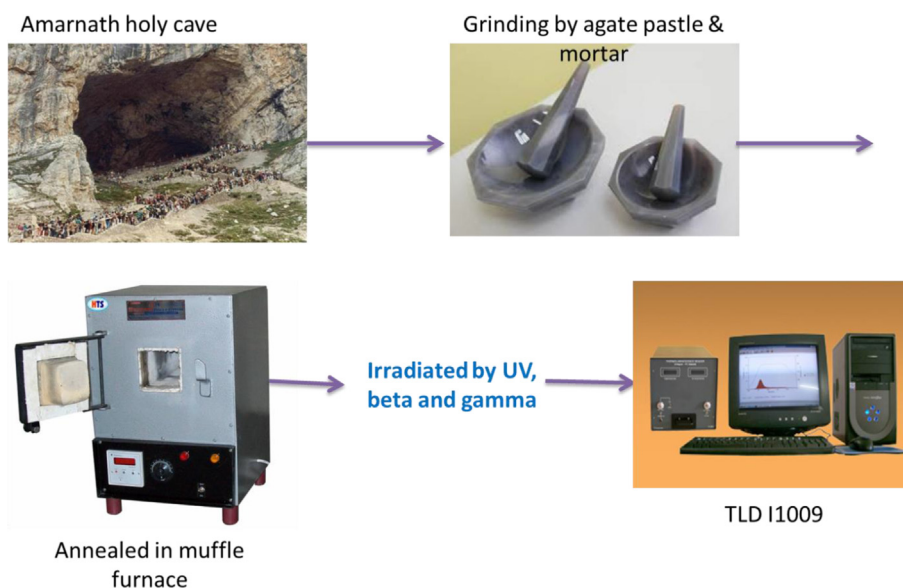


Fig. 1 – Process flow diagram of experimental study.

example the last heating of firestone as a result of human activity, the growth periods of corals (Gallois, Guegen N'Bechtel, & Schoerer, 1979) and mollusk shells (Ninagawa et al., 1992), and crystallization of calcite in carbonate deposits (Aitken & Bussel, 1982; Dubey, 2012; Dubey, Kaur, & Suryanarayana, 2010; Dubey, Kaur, Suryanarayana, & Murthy 2014; Hou, 1988; Tamrakar, Bisen and Brahme 2014a, 2014b; Tamrakar et al., 2013; Turetken et al., 1979).

Thermoluminescence dosimetry is a field of recent origin which has proved to be special importance in the estimation of radiation doses. Special attention has been paid to the development of materials for TL dosimetry. Number of scientists have standardized many materials for TL dosimetry and investigated in detail the fact that if luminescent material satisfies certain minimum requirements (dosimetric properties), they can be used in radiation dose estimation in TLD (Dubey et al. 2013; Tamrakar & Bisen, 2013; Tamrakar, Upadhyay, & Bisen, 2014; Tiwari, Kuraria, & Tamrakar, 2014).

2. Experimental

The natural limestone sample was collected from Amarnath holy cave. The TL glow curve was recorded by TLD Reader. The elemental analysis was done by EDXS technique. Irradiation source were used, UV (254 nm source), beta (Sr^{90}) and Gamma (Co^{60}). For TL measurement every time 2 mg weighed specimen is taken. From the data TL glow curve has been drawn using Origin 8 software and the shape factor (μ) has been calculated. The sample was characterized by XRD. The XRD measurements were carried out using Bruker D8 Advance X-ray diffractometer. The X-rays were produced using a sealed tube and the wavelength of x-ray was 0.154 nm (Cu K-alpha).

The X-rays were detected using a fast counting detector based on Silicon strip technology (BrukerLynxEye detector) (Dubey, Tiwari, Pradhan et al., 2014; Dubey, Tiwari, Tamrakar et al., 2014; Tamrakar, Bisen, & Brahme 2014a, 2014b; Tamrakar, Bisen, Sahu, & Brahme, 2014a, 2014b; Tamrakar, Bisen, Upadhyay, & Bramhe, 2014; Tamrakar, Bisen, Upadhyay, & Tiwari, 2014; Tamrakar, Kowar, Uplop, & Robinson, 2014; Tamrakar, Tiwari et al., 2015; Tamrakar, Upadhyay et al., 2015; Tiwari et al. 2014). Annealing the samples in different temperatures (Tamrakar, Bisen, & Bramhe, 2014a; Tamrakar, Bisen, Dubey et al., 2014; Tamrakar, Bisen, & Brahme, 2015):

- Samples pre annealed for 1 h at 200 °C, and immediately cooled to room temperature.
- Samples pre annealed for 1 h at 400 °C, and immediately cooled to room temperature.
- Samples pre annealed for 1 h at 600 °C, and immediately cooled to room temperature.
- Samples pre annealed for 1 h at 800 °C, and immediately cooled to room temperature.

Process flow chart of sample collection and experimental detail shown in Fig. 1.

3. Results and discussion

The XRD pattern of the sample is shown in Fig. 2. The width of the peak increases as the size of the Crystallite decreases. Broadened peak shows the nano crystalline behavior of the sample. The size of the crystal has been computed from the full width half maximum (FWHM) of the intense peak using Scherer's formula (Tamrakar, 2012). Crystallite size of sample

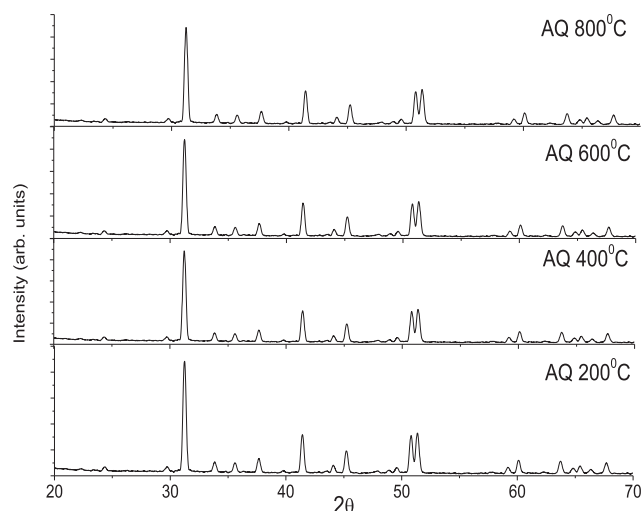


Fig. 2 – XRD pattern of limestone.

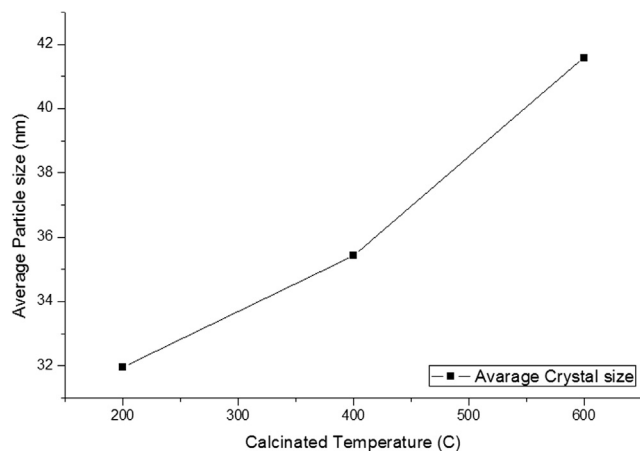


Fig. 3 – Calcinated temperature vs Crystal size of limestone.

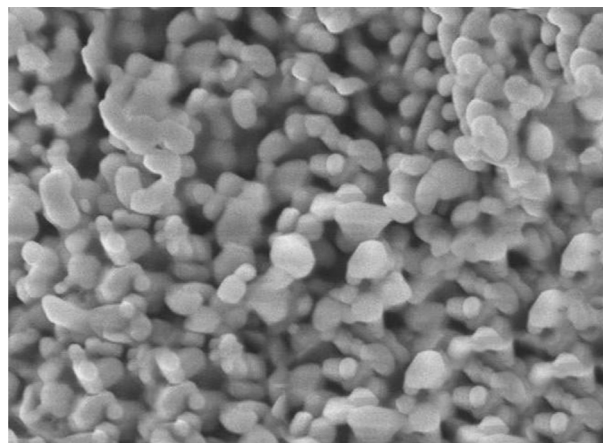


Fig. 4 – SEM image of limestone.

in the range 40 nm was found for 600 °C. Formula used for calculation is (Table 1).

$$D = \frac{0.9\lambda}{\beta \cos \theta}$$

Here D is Crystal size.

β is FWHM (full width half maximum).

λ is the wavelength of X ray source.

θ is angle of diffraction.

Fig. 3 shows the increasing Crystallite size with increases the annealing temperature of natural limestone. It is concluded that the temperature dependence of particle size.

3.1. Field emission gun scanning electron microscopy (FEGSEM)

The surface morphology of prepared phosphor was represented by FEGSEM images. From SEM images it is concluded that the prepared phosphor shows nanocrystalline behavior and good connectivity with grain which shows that powder size and morphology are well controlled. No significant difference is observed in XRD patterns and SEM micrographs (Fig. 4)

3.2. HRTEM (high resolution transmission electron microscopy)

Fig. 5 (a, b & c) displayed the HRTEM images of samples prepared under different resolutions. All these samples exhibited

Table 1 – Crystal size calculation of natural limestone (a) calcinated at 200 °C, (b) calcinated at 400 °C and (c) calcinated at 600 °C.

XRD analysis				
Prepared phosphor	2θ	FWHM in θ	Crystal size (nm)	Average crystallite size (nm)
Natural limestone calcinated at 200 °C	32.23	0.27	30.29	31.94
	41.42	0.25	33.60	
Natural limestone calcinated at 400 °C	32.14	0.25	32.70	35.42
	41.21	0.22	38.15	
Natural limestone calcinated at 600 °C	32.22	0.21	38.94	41.58
	41.56	0.19	44.23	

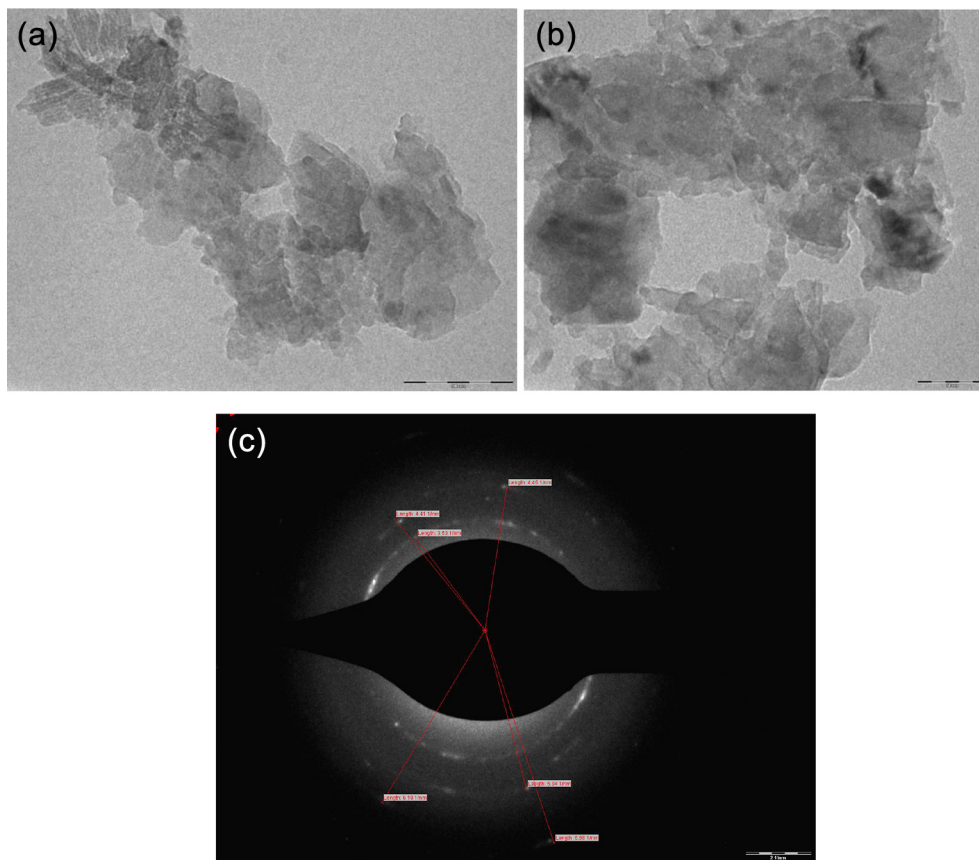


Fig. 5 – (a-c) TEM images of limestone.

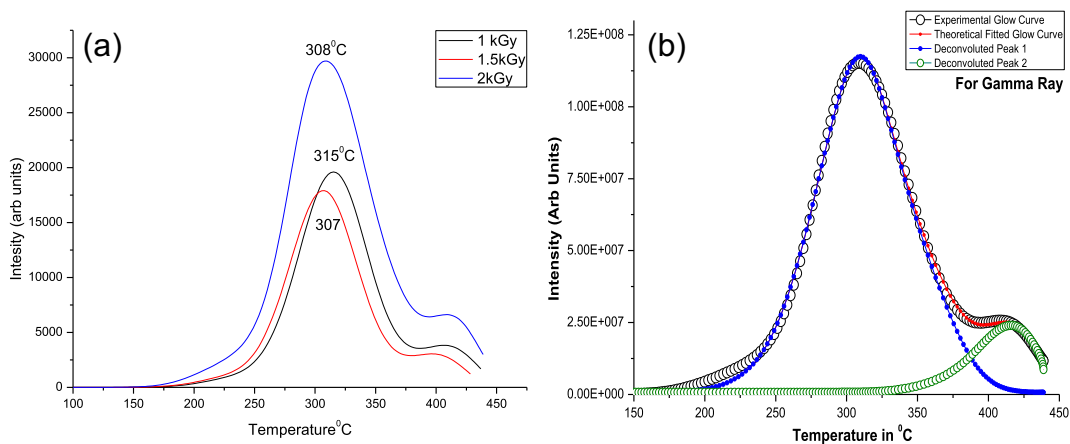


Fig. 6 – (a & b). Pure Amarnath stone gamma irradiated.

Table 2 – Kinetic parameters of Pure Amarnath stone gamma irradiated for different Gamma dose.

Dose in kGy	T ₁ (°C)	T _m (°C)	T ₂ (°C)	τ	δ	ω	μ = δ/ω	Activation energy E in eV	Frequency factor S in s ⁻¹
1	282	315	340	33	25	58	0.431	1.341	3.6 × 10 ¹²
1.5	274	307	340	33	33	66	0.5	1.322	3.9 × 10 ¹²
2	272	308	351	36	43	79	0.544	1.229	5.4 × 10 ¹¹

Table 3 – Typical trapping parameters of the deconvolution peaks, the sample was exposed for 2 kGy Gamma dose.

Peaks	T ₁ (°C)	T _m (°C)	T ₂ (°C)	μ _g	E (eV)	S (s ⁻¹)
Peak 1	286	309	337	0.549	1.929	9.3 × 10 ¹⁷
Peak 2	384	415	437	0.415	1.958	2.9 × 10 ¹⁵

Table 5 – Typical trapping parameters of the deconvolution peaks. The sample was exposed for 2 kGy Gamma dose.

Peaks	T ₁ (°C)	T _m (°C)	T ₂ (°C)	μ _g	E (eV)	S (s ⁻¹)
Peak 1	288	314	339	0.49	1.718	9.1 × 10 ¹⁵
Peak 2	289	335	381	0.5	1.04	3.7 × 10 ⁰⁹

a sphere-like morphology with the particle size of about 35–47 nm. This two-dimensional growing habit coincided with the concept of preferential nucleation in this system, when the particle size continued to increase, a regular morphology of hexagonal flake was also observed, indicating that the crystals were better crystallized. This images are very good agreement with XRD and FEGSEM results. It is clearly shows the formation of nanosphere.

3.3. Thermoluminescence glow curve analysis

The behavior displayed by TL glow curve of natural limestone irradiated by γ-dose with 1 kGy, 1.5 kGy and 2 kGy (Fig. 6a) having good intensity. The TL glow curve intensity increases with increasing the dose of gamma radiation. It shows linear response with dose means that the natural limestone is good TL material. The peak temperature for TL glow curve found at 308 °C with a shoulder peak at high temperature it shows the less fading and high stability of the limestone sample. Here the high temperature peaks also indicates that the formation

of deep trapping in the sample and the experimental TL glow curve fitted by CGCD techniques (Fig. 6b). From CGCD technique suitable glow curve found for the calculation of kinetic parameters for dual peak in TL glow curve. It is very difficult to determine the peak values and shape factor for dual TL peak so the CGCD technique is useful for the calculation (Kitis, Gomez Ros, & Tuyn, 1998; Tamrakar, Tiwari et al., 2015; Tamrakar, Bisen, & Brahme, 2014a, 2014b; Tamrakar, Bisen, Sahu, & Brahme, 2014a, 2014b). (Tables 2 and 3).

Finding an optimum pre-irradiation annealing regime for TL materials is of crucial importance in TL dosimetry. Therefore, the effects of annealing temperature and duration on TL characteristics of the natural limestone were studied. It was found that the TL sensitivity of the produced nanoparticles vary for different applied annealing regimes. The optimum sensitivity was realized for thermal treatment of 200–400 °C for 60 min (Fig. 7a) and this annealing regime was used for succeeding studies. In order to identify the number of glow peaks contained in the complex glow curve of the natural stone, CGCD technique was employed. The difference

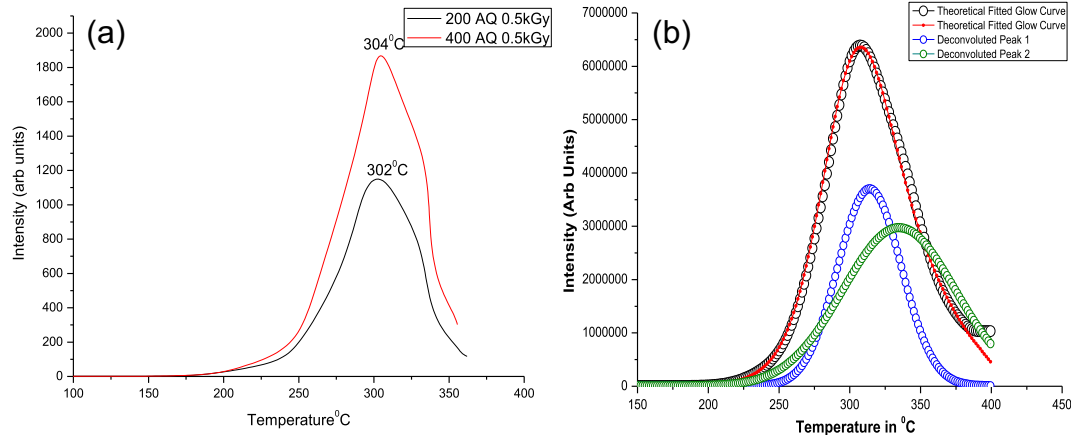


Fig. 7 – (a & b). Amarnath stone gamma AQ effect for beta irradiation.

Table 4 – Kinetic parameters of Amarnath stone gamma AQ effect for fixed beta irradiation for different annealing temperature with heating rate 6 °C/s.

Annealing temperature (°C)	T ₁ (°C)	T _m (°C)	T ₂ (°C)	τ	δ	ω	μ = δ/ω	Activation energy E in eV	Frequency factor S in s ⁻¹
200	274	302	334	28	32	60	0.533	1.543	5.1 × 10 ¹⁴
400	281	304	341	23	37	60	0.617	1.924	1.2 × 10 ¹⁸

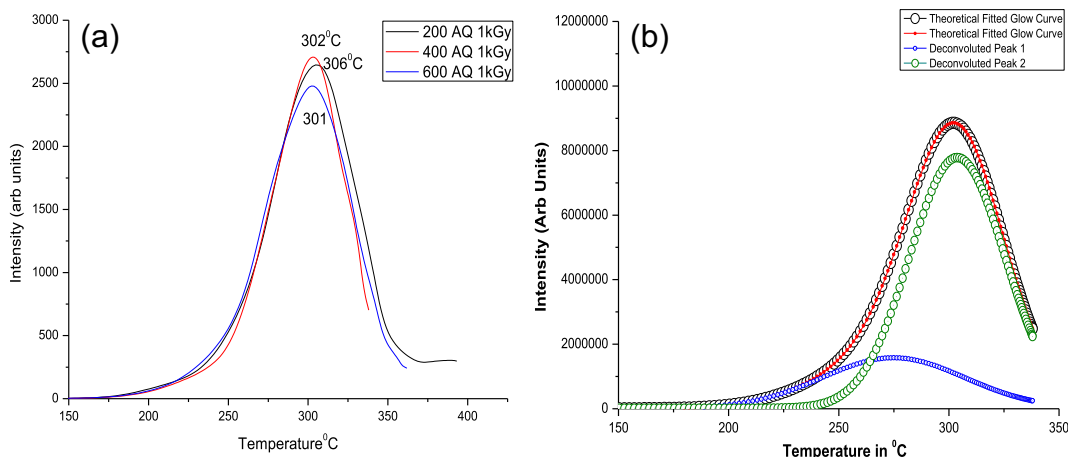


Fig. 8 – (a & b) For 1 kGy irradiation experimental glow curve and fitted glow curve of natural limestone.

Table 6 – Kinetic parameters of 1 kGy irradiation of natural limestone.

Annealing temperature (°C)	T ₁ (°C)	T _m (°C)	T ₂ (°C)	τ	δ	ω	μ = δ/ω	Activation energy E in eV	Frequency factor S in s ⁻¹
200	273	306	337	33	31	64	0.484	1.313	3.4 × 10 ¹²
400	272	302	329	30	27	57	0.474	1.423	4.1 × 10 ¹³
600	268	301	328	33	27	60	0.45	1.282	2.3 × 10 ¹²

Table 7 – Typical trapping parameters of the deconvolution peaks. The sample was exposed for 2 kGy Gamma dose.

Peaks	T ₁ (°C)	T _m (°C)	T ₂ (°C)	μ _g	E (eV)	S (s ⁻¹)
Peak 1	235	274	313	0.5	0.993	1.5 × 10 ¹⁰
Peak 2	272	303	329	0.456	1.377	1.5 × 10 ¹³

between the intensity of the experimental glow curve and that obtained by adding the intensities of the fitted glow peaks is shown as residue at the top of the Fig. 7b.

Glow peaks were deconvolved using CGCD program, which was produced in our laboratory using the Levenberg–Marquart algorithm. This program uses the general order kinetics, the mixed order kinetics and complex functions describing the continuous trap distribution to parameterize the shape of the glow peaks. The TL intensity in general order kinetics model in terms of the intensity and the

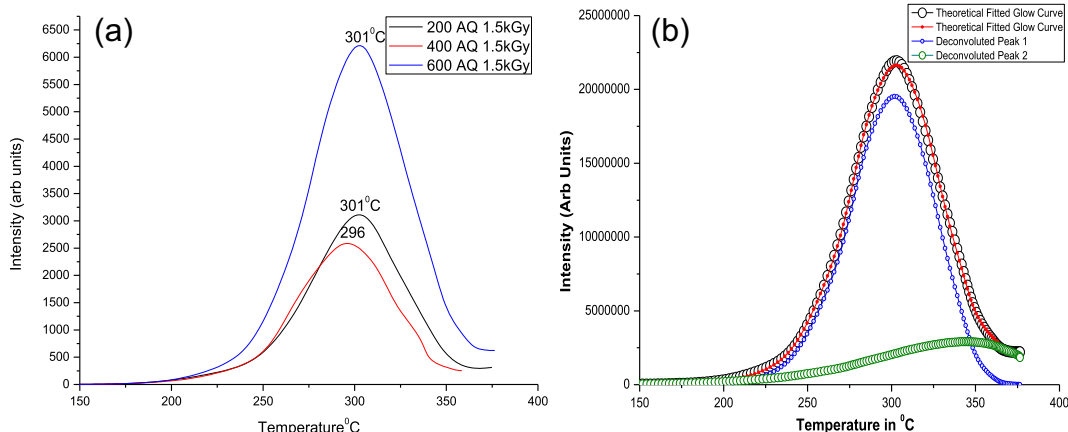


Fig. 9 – (a & b) Gamma 1.5 kGy irradiated annealed limestone.

Table 8 – Kinetic parameters of 1 kGy irradiation of natural limestone.

Annealing temperature (°C)	T ₁ (°C)	T _m (°C)	T ₂ (°C)	τ	δ	ω	μ = δ/ω	Activation energy E in eV	Frequency factor S in s ⁻¹
200	270	300	332	30	32	62	0.516	1.425	4.8 × 10 ¹³
400	263	296	327	33	31	64	0.484	1.268	2.1 × 10 ¹²
600	271	301	334	30	33	63	0.524	1.432	5.3 × 10 ¹³

Table 9 – Typical trapping parameters of the deconvolution peaks. The sample was exposed for 2 kGy Gamma dose.

Peaks	T ₁ (°C)	T _m (°C)	T ₂ (°C)	μ _g	E (eV)	S (s ⁻¹)
Peak 1	272	302	330	0.483	1.425	1.5 × 10 ¹⁰
Peak 2	281	339	378	0.402	0.809	4.3 × 10 ¹³

temperature of the peak maximum is given by [29]. For annealed sample of limestone shows the TL glow peaks at 304 °C and the fitted glow curve for beta irradiation (Fig. 7 a&b). (Tables 4 and 5).

It may also be noted that the TL glow peak temperature and intensity depend on various parameters such as the nature of the sample – crystalline, semi-crystalline, and amorphous, the history of the sample (pre-exposed to natural radiations, age of the sample, etc.), impurity content, heat treatment given to the sample prior to irradiation, the nature of the ionizing radiation, the amount of irradiation (dose), temperature at which the TL measurements are made, the time interval between the measurements, the environment of the sample during experiment (humidity, atmospheric gas, etc.), the type of detector and the heating rate [30].

Here the TL glow curve analyzed for UV irradiated sample of natural limestone for optimized heating rate 5 °C s⁻¹ and three different annealing temperatures 200, 400 and 600 °C for 1 kGy irradiation. It shows well resolved peak at 302 °C (Fig. 8a) and the fitted glow peaks shows good TL glow fit for experimental glow curve (Fig. 8b). It implies that creations of trapping centers were low for lower doses and high for higher doses. This behavior of the sample is useful for dosimetric application. Each of these TL glow curves are analyzed based on glow curve shape method modified by Chen's (Chen & Kirsh, 1981; Tamrakar, Bisen, & Brahme, 2014a; Tamrakar et al., 2014b; Tamrakar, Bisen, Sahu, & Brahme, 2014a, 2014b). It is very clear from above TL glow peaks for beta and gamma irradiation the formation of more traps and the high temperature peaks were found for beta and gamma irradiated limestone but for UV irradiation the surface traps formation shows lower temperature peaks. (Tables 6 and 7).

For the optimized gamma dose 1.5 kGy and optimized heating rate 5 °Cs⁻¹ the TL glow curve recorded with the variation of annealing temperature 200, 400 and 600 °C show good TL glow peaks at 300 above temperature (Fig. 9a) which shows the formation of deep trapping and the corresponding kinetic parameters are calculated by peak shape method after

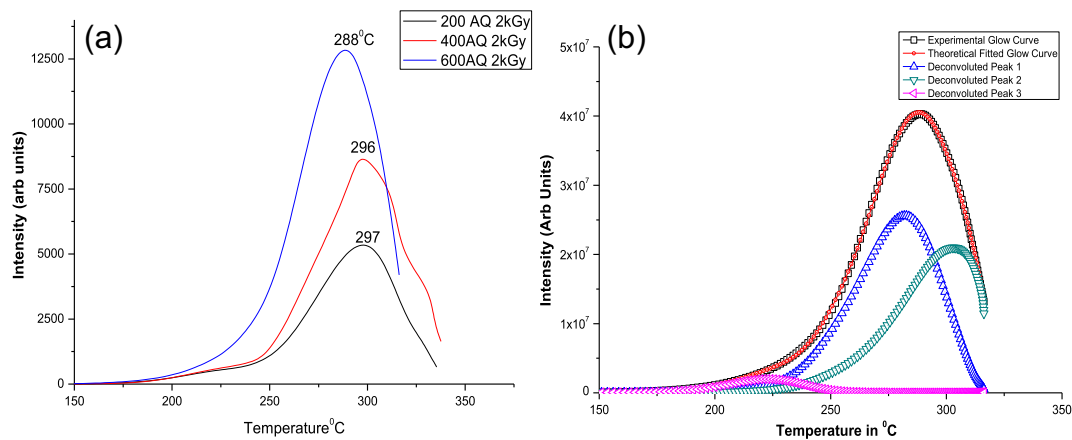


Fig. 10 – (a & b) Gamma (2kGy) irradiated annealed limestone.

Table 10 – Kinetic parameters of 1 kGy irradiation of natural limestone.

Annealing temperature (°C)	T ₁ (°C)	T _m (°C)	T ₂ (°C)	τ	δ	ω	μ = δ/ω	Activation energy E in eV	Frequency factor S in s ⁻¹
200	269	297	320	28	23	51	0.451	1.494	2.4 × 10 ¹⁴
400	265	296	325	31	29	60	0.483	1.35	1.2 × 10 ¹³
600	260	288	312	28	24	52	0.462	1.449	1.5 × 10 ¹⁴

Table 11 – Typical trapping parameters of the deconvolution peaks. The sample was exposed for 2 kGy Gamma dose.

Peaks	T ₁ (°C)	T _m (°C)	T ₂ (°C)	μ _g	E (eV)	S (s ⁻¹)
Peak 1	257	282	300	0.419	1.581	3.7 × 10 ¹⁵
Peak 2	275	302	319	0.386	1.563	7.7 × 10 ¹⁴

fitting the experimental TL glow curve by CGCD techniques (Fig. 9b). (Tables 8 and 9).

Fig. 10 (a) shows TL glow curve of gamma (2kGy) irradiated lime stone with the variation of annealing temperature 200–600 °C. It shows good TL glow curve at 288 °C intense peak found for 600 °C annealing. The deconvoluted peak which is fitted theoretically with experimental glow curve, two distinct peaks were found. Fig. 10 (a & b) is here. (Tables 10 and 11).

Here the pure as collected amarnath stone (lime) shows good TL glow curve at 314 °C with shoulder peak at 399 °C (Fig. 11a) and CGCD fitted peaks (Fig. 11b). (Tables 12 and 13) (Tamrakar and Dubey, 2014).

3.4. Chemical composition by EDXS study

The chemical composition analysis of limestone was determined by EDXS study and major components are Ca, O, Al, C found so from the analysis it is confirmed the sample was limestone collected from the Amarnath holy cave (Fig. 12). (Table 14).

4. Conclusion

1. The sample of natural stone was collected from Amarnath holy cave and the elemental analysis (EDXS) confirms it is limestone.

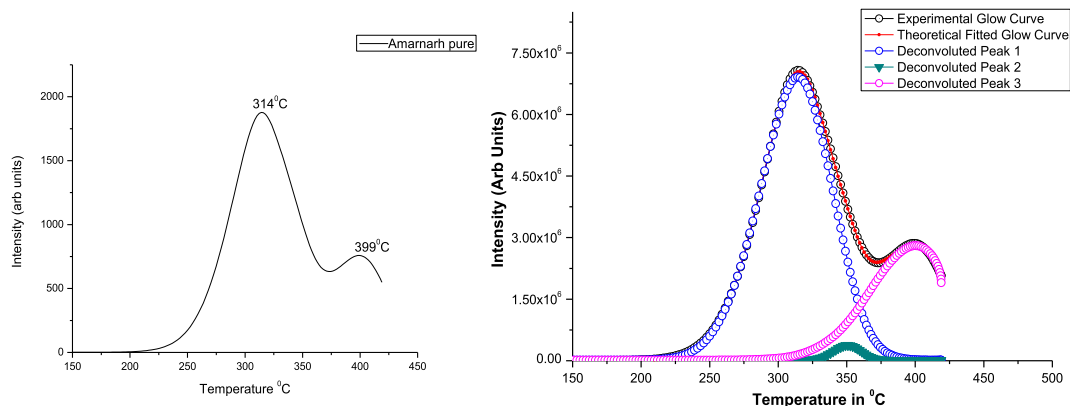


Fig. 11 – (a & b) Pure as collected limestone from Amarnath holy cave.

Table 12 – Kinetic parameters of 1 kGy irradiation of natural limestone.

Type	T ₁ (°C)	T _m (°C)	T ₂ (°C)	τ	δ	ω	μ = δ/ω	Activation energy E in eV	Frequency factor S in s ⁻¹
Pure Stone	283	314	352	31	38	69	0.551	1.459	4.5 × 10 ¹³

Table 13 – Typical trapping parameters of the deconvolution peaks. The sample was exposed for 2 kGy Gamma dose.

Peaks	T ₁ (°C)	T _m (°C)	T ₂ (°C)	μ _g	E (eV)	S (s ⁻¹)
Peak 1	282	313	345	0.508	1.44	3.2 × 10 ¹³
Peak 2	358	401	430	0.403	1.342	1.1 × 10 ¹¹

2. Collected sample were characterized by XRD, SEM, TEM technique and Crystallite size calculated by Scherer's formula by XRD techniques and it is in nano range.
3. The as collected sample annealed in different temperature 200, 400 and 600 °C for 1 h and the particle size increase with increasing the annealing temperature.
4. The as collected sample were exposed in UV, beta and gamma radiations and corresponding TL glow curve are recorded for different exposure as well as different annealing temperature.
5. The kinetic parameters are calculated by CGCD technique for every fitted and experimental curve.
6. Here the high temperature TL glow curve shows less fading and high stability and its reproducibility is also high.
7. The corresponding activation energy is also high for every TL glow curve its ranges are 1.3–1.68 eV.
8. Maximum TL glow curve shows the general and second order of kinetics.
9. It shows formation of deep trap in the as collected sample and it is high for pure sample which exposed naturally.
10. The CGCD technique is best for the calculation of kinetic parameters in TL glow curve.

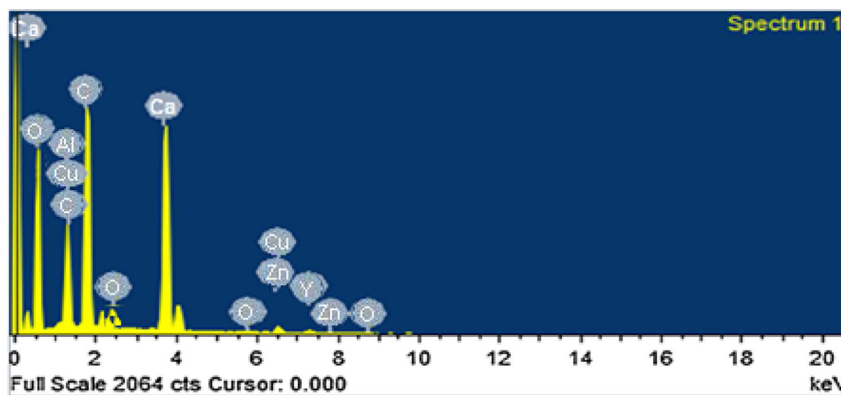


Fig. 12 – EDX image of as collected limestone from Amarnath holy cave.

Table 14 – EDX of as collected limestone from Amarnath holy cave.

S. no	Element	Weight%	Atomic%
1.	C K	30.17	52.45
2.	O K	29.25	38.18
3.	Ca K	32.78	7.70
4.	Cu K	1.61	0.53
5.	Zn K	1.23	0.39
6.	Y L	0.18	0.10
7.	Al L	4.77	0.66
Totals		100.00	

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