AN ATTEMPT TO CORRECT FOR THE FADING
IN MILLION YEAR OLD BASALTIC ROCKS

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Abstract: The use of feldspar for luminescence dating has been restricted because of anomalous fading. This has made its application to several important geological problems such as volcanic terrains difficult. Presently, two correction procedures are used to correct for anomalous fading. The present study tests these correction procedures using volcanic samples of known ages spanning the time period of 400 ka to 2.2 Ma. These correction procedures provided grossly underestimated ages (up to 60%). The possible causes for the underestimation are discussed.

Keywords: Anomalous fading, fading correction, dating of basaltic rocks.

1. INTRODUCTION

Anomalous fading is ubiquitous in feldspar minerals and leads to undesirable loss of trapped charge, causing an underestimation of ages. This restricts the use of feldspar as a natural dosimeter for chronological purposes both in terrestrial and in extraterrestrial settings. In the latter, often feldspar is the only luminescent phase. There have been considerable attempts to either circumvent or correct for the fading loss in nature. The first attempt was made by Huntely and Lamothe (2001) and they have demonstrated that the correction is possible for the samples below 20-50 ka. More recently a new mathematical expression was published by Huntley (2006) to describe the loss in signal due to anomalous fading after an instantaneous irradiation. Using this, Kars *et al.* (2008) pro-

posed a correction procedure that was successful in predicting the field saturation dose in an infinitely old sample, and could provide correction for ages up to 325 ka. This contribution examines these fading correction procedures on basalt samples of known ages.

2. SAMPLES AND EXPERIMENTAL DETAILS

Eight volcanic samples were studied from Flores (FL) and Fayal (FA) Islands, Azores, Portugal. The rocks correspond to the basalt and hawaiite types in the age range of 400 ka to 2.2 Ma. The existing chronology is based on volcano-stratigraphic methods and correlations with previous radiometric determinations on equivalent samples (see details in Azevedo and Portugal Ferreira, 2006). The details of these samples are summarized in **Table 1**. Samples for luminescence measurements were prepared after removing the outer layer of the collected

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Table 1. *Tabulated values of measured D$_e$, dose rates and age estimates for 8 basaltic rock samples. Expected ages are also given.*

Sample	D_e (Gy)	Dose rate (Gy·ka^{-1})	Age (ka)	Expected age[*] (ka)
FL2	161±3	2.6±0.1	62.1±2.6	1000-1500
FL3	69.9±1.9	3.0±0.1	23.0±1.0	400
FL4	149.2±3.7	2.7±0.1	55.3±2.5	2000-2200
FL5	88.9±1.2	3.0±0.1	29.6±1.2	500-670
FL6	68.2±1.2	0.8±0.1	85.2±4.5	400
FL10	166.8±1.9	3.2±0.1	52.1±1.7	670
FA2	56.5±2.3	1.6±0.1	35.3±2.6	< 1000
FA5	1.7±0.7	2.3±0.1	0.7±0.3	< 1000

[*]*Based on volcano-stratigraphic methods and correlations with previous radiometric determinations on equivalent samples (Azevedo and Portugal Ferreira, 2006).*

rock pieces in the dark by sawing and thereafter crushing the inner material. The size fraction 150-210 µm was then obtained by sieving and used for luminescence measurements without any further chemical treatment.

The light exposed part of the rock materials were crushed further to make it powder like. These powder samples were used to estimate the elemental concentration of U, Th and K using gamma spectrometry with NaI(Tl) crystal detector. The alpha efficiency (*a*) value of 0.1 was assumed in order to calculate the alpha dose contribution. Using the dose and dose rate values, the age estimates were derived and they are given in **Table 1**.

The measurements were carried out using a Risø TL/OSL-DA-15 reader. Blue light stimulation used an array of LEDs (470±30 nm) filtered through GG-420 long-pass filters, and delivering ~50 mW·cm^{-2} at the sample position. IR stimulation was carried out using IR LEDs (870±40 nm) with a maximum intensity of ~150 mW·cm^{-2}. Calibrated beta sources (^{90}Sr/^{90}Y) delivering between 0.22 (Risoe 1), 0.11 (Risoe 2) and 0.057 (PRL) Gy·s^{-1} were used to irradiate the samples in the reader. An EMI 9635QB photomultiplier tube with bialkali photocathode was used. Blue emission (320-460 nm) was detected through a 4 mm Corning 7-59 and 2 mm Schott BG-39 optical filter combination. UV emission (280-380 nm) was detected through 7.5 mm of Hoya U-340 filter.

3. MEASUREMENT PROTOCOLS

The D_e values were measured by single aliquot regenerative (SAR) procedure (Murray and Wintle, 2000) where a test dose induced luminescence signal was used to correct the possible sensitivity changes. The details about the preheat and the stimulation temperatures are given in the subsequent sections. The fading measurements were carried out using SAR procedure where the luminescence signals of prompt and various time delays were measured (Auclair *et al*., 2003). The fading rates (*g*-values in %/decade) were calculated by fitting **Eq. 3.1** to the measured data of sensitivity corrected luminescence vs. time delay, t_d,

$$I = I_0 \left[1 - g \log\left(\frac{t_d}{t_c}\right) \right] \qquad (3.1)$$

where I and I_0 are the sensitivity corrected luminescence at t_d (time delay) and t_c (prompt) respectively. The *g*-values are standardized to t_c = 2 days.

4. SIGNAL SELECTION

In the present sample conventional blue emission under IR stimulation at 50ºC (OSL$_{IR-B}$ [50ºC]), was close to the limits of detection and hence was not used. Then blue emission under IR stimulation at elevated temperature (200ºC) was measured (Tsukamoto *et al*., 2011). The decay curves of this signal of samples FL4 and FL5 are shown in **Fig 1**. The arithmetic mean of the measured D_e values of FL4 and FL5 are 170±26 Gy and 3±6 Gy respectively. Using a test dose error (20%) and recycling ratio (0.8-1.2), only 8 out of 12 aliquots were accepted in case of sample FL4. For FL5 the number of acceptable results was 6 out of 12 aliquots. The arithmetic means of *g*-values of FL4 and FL5 are 16±4 and 17±2%/decade,
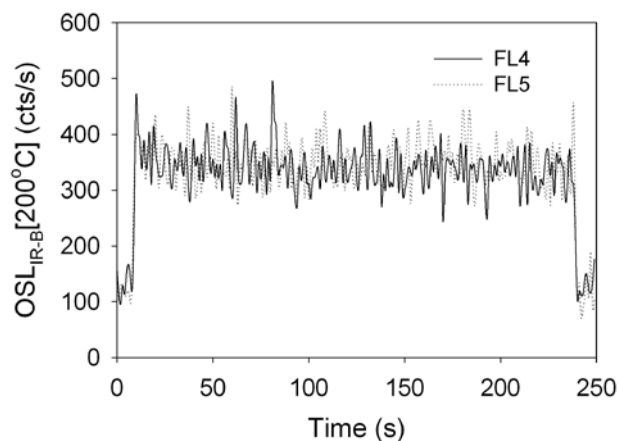

Fig. 1. *OSL$_{IR-B}$ measured at 200ºC is shown for two samples, a) FL4 and b) FL5.*

respectively. Because of low signal intensity and large *g* values, the OSL_{IR-B} [200ºC] was not used for age calculation.

We then investigated the OSL_{B-UV}[50ºC] signal with a preheat of 250ºC at 2ºC/s for 60 s. The natural and 220 Gy beta dose induced luminescence signals of FL4 are shown in **Fig. 2**. The natural signal was undetectable and hence this signal was not considered for age calculation. Then the OSL_{B-UV}[225ºC] signal with a preheat of 250ºC at 2ºC/s for 150 s was explored. The measured D_e values of FL2, FL3, FL4, FL5, FL6 and FL10 are 22.2±0.4, 8.1±0.4, 25±1, 13±1, 7±1 and 18±1 Gy respectively. All the values are the arithmetic mean of 4 aliquots for each sample. The D_e values of FA2 and FA5 were 11.5±0.4 and 0.8±0.2 Gy using 12 aliquots each. The arithmetic mean of the measured *g*-values is 23.5±1.3%/decade for all the 8 samples. The widely used correction procedure (Huntley and Lamothe, 2001) does not work for such high fading values (Morthekai *et al.*, 2008) as its use results in negative intensity in a relatively shorter time scales and hence put a limit for age correction. Hence this signal was also not considered.

Earlier studies have shown a TL_{UV} signal around 525ºC in the basaltic materials (Guerin and Valladas, 1980) and that could be bleached by blue light (Morthekai *et al.*, 2008). We explored this signal further. Some of its characteristics are mentioned elsewhere (Morthekai *et al.*, 2008). It comprises two TL glow peaks at 525ºC and 600ºC (heating rate, 2ºC/s) after an additional laboratory dose and a preheat of 380ºC for 150 s. The 525ºC peak could be bleached by blue light but not the 600ºC peak (**Fig. 3**). For the rest of this study, the OSL (blue stimulation) signal at 360ºC, OSL_{B-UV} [360ºC], was used in this study for age calculation.

5. AGE CALCULATION

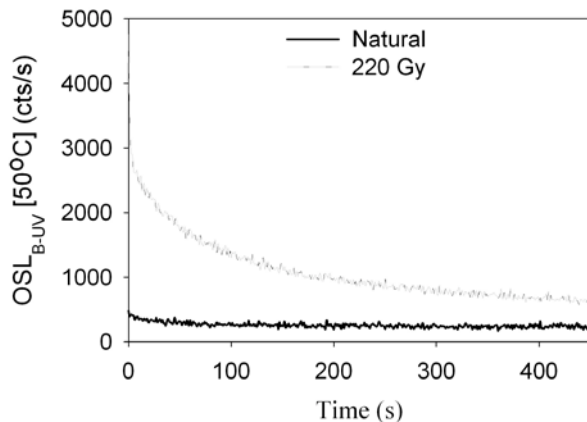The palaeodoses of each sample were measured using single aliquot regenerative (SAR) dose protocol (Murray and Wintle, 2000). Samples were preheated to 380ºC at the heating rate 2ºC/s and kept for 150 s to reduce the ITL at the stimulation temperature i.e., 360ºC. Recycling ratio was in the range of 0.9-1.1 and the test dose error was within 10%. The sensitivity corrected natural OSL signal was interpolated into the single saturated exponential dose response curve, DRC, to get the palaeodose. Twelve aliquots were measured for each sample. Using the dose and dose rate values, the age estimates were derived and they are given in **Table 1**. Except FA2 and FA5, the ages are only 10% of the expected ages and the underestimation might be because of anomalous fading in OSL_{B-UV} [360ºC] also.

6. FADING CORRECTION

The fading of OSL_{B-UV} [360ºC] signal was measured for all the 8 samples and the fading was corrected using two different procedures (CP):

CP1: The correction procedure of Huntley and Lamothe, 2001 and

CP2: The correction procedure of Kars *et al.*, 2008 and Kars and Wallinga, 2009.

CP1 is based on the understanding that the anomalous fading follows logarithmic decay with time while CP2 assumed that it depends on the number density of luminescence centres and the fading follows $\exp[-\rho' \ln(s\,t)^3]$ with time where ρ' is the fading parameter related to the number density of luminescence centres in the crystal and *s* is the attempt-to-escape frequency factor in s^{-1} (Huntley, 2006). The details of the procedures are given in the subsequent sections.

Correction procedure CP1

This is widely used procedure and is based on three assumptions, viz.,

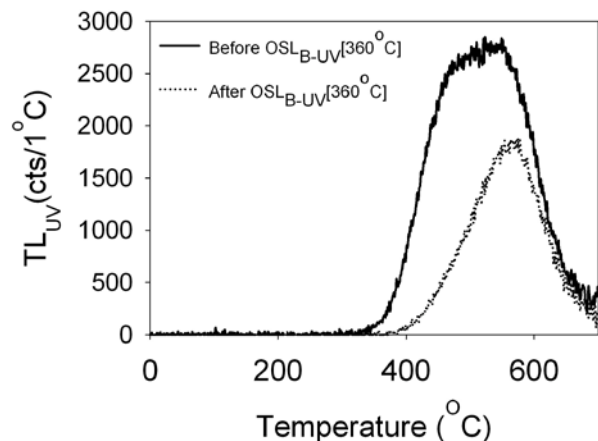1) trapped charges are received by the crystal at constant rate due to irradiation,

Fig. 2. *OSL_{B-UV} signals, both natural and laboratory irradiated, measured at 50ºC temperature, are shown.*

Fig. 3. *Thermoluminescence signals peak at 525ºC and 600ºC at 2ºC/s after an additional laboratory dose and a preheat of 360ºC for 150 s before and after blue light stimulation for 500 s.*

2) every increment of trapped charges recombine independent of earlier increments and

3) luminescence signal fades logarithmically with time.

The implication of the first and second assumptions is that the natural luminescence lies in the linear part of the DRC. The third assumption predicts negative luminescence intensity after a certain time since the cessation of irradiation and limits the correction beyond that time.

With the exception of FL4, the condition based on first and second assumptions was met in all samples. The measured dose responses and the natural sensitivity corrected luminescence signal, L/T for FL2, FL4 and FL10 are shown in **Fig. 4**.

In CP1, as per the third assumption, the sensitivity corrected luminescence signals (L/T) is plotted against $\log[t_d]$ where t_d is delay time since the cessation of laboratory irradiation which is roughly the sum of half of the irradiation time and time taken to preheat (Auclair *et al.*, 2003). Then the data is fitted with **Eq. 3.1** (**Fig. 5**) and the fading parameter, g, is derived from the slope.
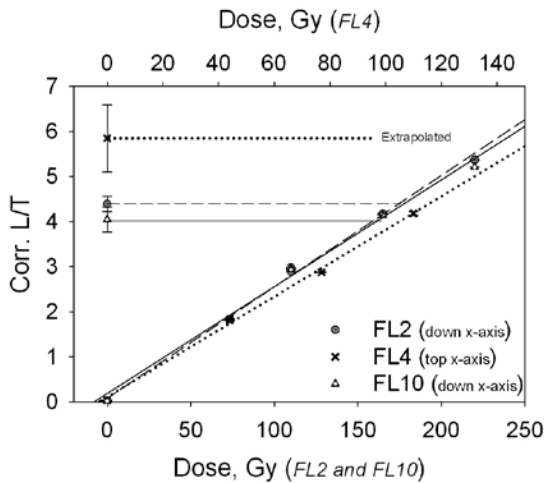
The correction formula (**Eq. 6.1**) which is the integration of the fading signals throughout the irradiation by assuming each increment of trapped charges is decaying independent of each other and the linearity between I and t with the rate I_0/T (first and second assumptions) is given as

$$T_f = T\left[1 - k\left\{\ln\left(\frac{T}{t_c}\right) - 1\right\}\right] \qquad (6.1)$$

where T_f and T are the measured and corrected ages and $k=g/(100 \cdot \ln[10])$ (Aitken, 1985, Huntley and Lamothe, 2001). Thus obtained ages are given in **Table 2**. The corrected ages are in gross under-estimation of 80% even though the conditions to apply this procedure are met.

Correction procedure CP2

This procedure by Kars *et al.* (2008) and Kars and Wallinga (2009) is based on Huntley (2006). The fading parameter, ρ' is obtained from the **Eq. 6.2** by fitting to the fading measurements. One such fitting for the sample FL4 is given in **Fig. 6** (thick line).
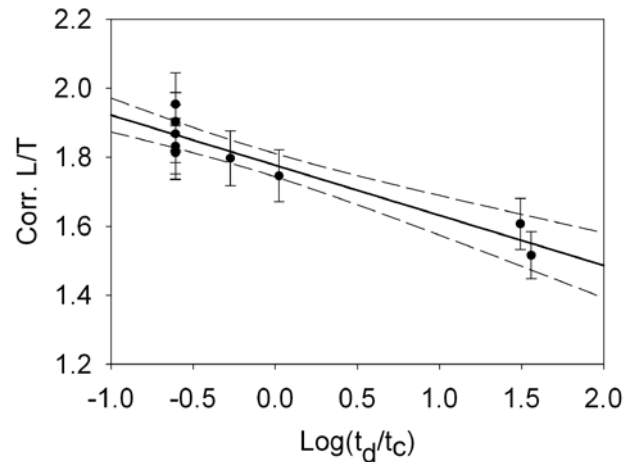

Fig. 4. *Measured dose response curves of FL2, FL4 and FL10. Except FL4, natural L/T points are well bracketed by the regenerative dose points.*

Fig. 5. *Fading measurements of FL4 and fitting of **Eq. 3.1** to the data and t_c is taken to be 1 hour.*

Table 2. *Tabulated values of uncorrected and corrected age estimates by CP1 for all the samples. Fading parameter, g-value is standardized to t_c = 2 days.*

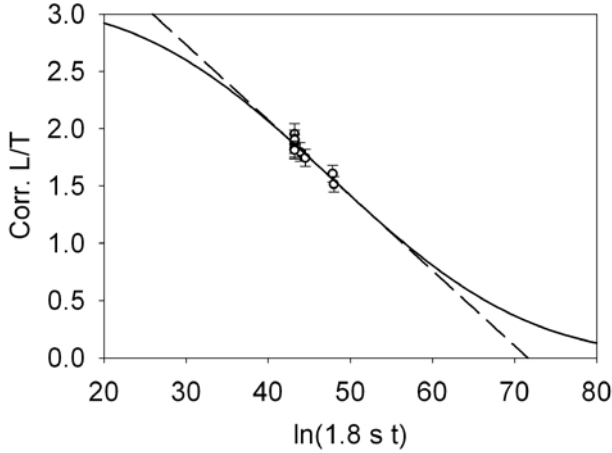| Sample	Apparent age (ka)	g-value (%/dec)	Delay time* (days)	Corrected age_CP1 (ka)	Expected age (ka)
FL2	62.1±2.6	10.2±0.7	6	219.8±12.5	1000-1500
FL3	23.0±1.0	8.1±0.9	6	46.7±3.4	400
FL4	55.3±2.5	9.5±0.8	84	138.4±9.4	2000-2200
FL5	29.6±1.2	9.3±0.5	84	65.8±4.1	500-670
FL6	85.2±4.5	7.7±0.8	5	181.8±16.3	400
FL10	52.1±1.7	8.4±0.6	5	119.9±6.2	670
FA2	35.3±2.6	9.8±2.7	12	98.3±11.3	< 1000
FA5	0.7±0.3	11.4±0.9	14	1.5±1.2	< 1000

*t_c used in **Eq. 6.1**.

Fig. 6. *Obtaining fading parameters (g and ρ') by fitting **Eq. 3.1** (as in **Fig. 5**) and **Eq. 6.3** to the fading measurements of FL4. Both functions fit very well in that limited measurable time scales in the laboratory.*

$$I = I_0\, e^{-\rho'\ln(1.8st)^3} \tag{6.2}$$

where I and I_0 are the intensity of luminescence signals at time t and at immediately after the irradiation and s is the attempt to escape factor assumed to be $3.0 \cdot 10^{15}$ s^{-1}. The correction is done as follows,

1) the natural OSL signal and the dose response curve with three or more regenerative points are measured
2) each of regenerative OSL points are corrected for the fading during the laboratory irradiation using **Eq. 6.2** with t (s) as $0.5 \cdot t_R + 550$ where t_R is the regeneration beta dose exposure time and 550 s is the time delay from irradiation to prompt measurement.
3) this corrected dose response curve is fitted to a single saturating exponential to get the saturating limit I_0 of the sensitivity corrected OSL and D_0, the onset of saturation
4) construct a dose response curve with a natural dose rate assuming I_0 is the limit for the OSL and the corresponding trapped charges are distributed in the crystal with the distance distribution with respect to the recombination centres as described by **Eq. 6.3** given below

$$p(r')dr' = 3r'^2\, e^{-r'^3}\, dr' \tag{6.3}$$

where r' is the dimensionless distance variable (Eq. 3 of Huntley, 2006). The r'_{av} and r'_{max} are calculated to be 0.893 and 0.874 respectively (**Fig. 10a**) and the derivations are given in Appendix. The issues of fading during irradiation and the evolution of different types of traps (250 types in this case) throughout the natural irradiation were taken care of (see Kars and Wallinga, 2009 for more details).

5) the measured natural OSL is to be interpolated into this constructed dose response curve which mimics the same during natural irradiation.

The fading parameter, ρ', of all the samples were derived and the arithmetic mean and standard deviation is $(5.63\pm0.83) \cdot 10^{-6}$. As it is required to have full dose response curve for the calculation, that of FL4 was measured with the intention to use it as a standardized response curve. The standardized curves of 4 samples are shown in **Fig. 7** and all the L/T measurements were corrected for the fading that occurred during the test dose irradiation like the step 2 above (except the t_R is replaced by t_{TD}, time to give test dose). The measured full response
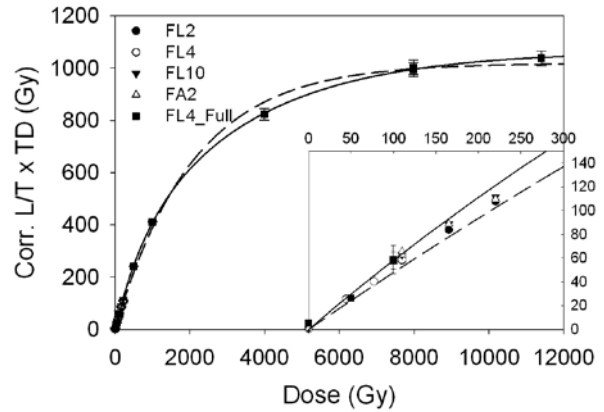

Fig. 7. *Standardized dose response curves of the FL2, FL4, FL10 and FA2 and one full dose response curve of FL4. They are fitted with single (dashed line) and double (thick line) saturating exponentials. Inset shows the form of the dose response curve is same for all the samples.*

Table 3. *Tabulation of uncorrected and corrected age estimates by CP2.*

Sample	Apparent age (ka)	Corrected age$_{CP2}$ (ka)	Expected age (ka)	Under-estimation (%)
FL2	62.1±2.6	407±22	1000-1500	65
FL3	23.0±1.0	168±15	400	54
FL4	55.3±2.5	350±57	2000-2200	80
FL5	29.6±1.2	193±6	500-670	64
FL6	85.2±4.5	157±46	400	49
FL10	52.1±1.7	362±33	670	38
FA2	35.3±2.6	124±8	< 1000	< 73
FA5	0.7±0.3	5±1	< 1000	< 98

of FL4 has been used for further calculations. The natural L/T points for all the samples were also corrected for test dose and fading during test dose irradiation. The test dose correction was necessary because the test dose for natural L/T and the regeneration L/Ts were different. The test dose for natural L/Ts was 33 Gy (for FL4 and FL5, that was 22 Gy) and that for regeneration L/Ts was 85.3 Gy. This standard response curve was corrected for fading during regeneration doses, as mentioned in step 2, using average ρ'. Using that the natural dose response curve was constructed and here also the average ρ' was used. Dose estimates were made by interpolating the corrected L/T points into the constructed standardized natural dose response curve. The measured dose response curve, unfaded dose response curve and the constructed natural dose response curves along with the highest natural L/T are shown in **Fig. 8** and the computed ages are given in **Table 3**. The associated error with the corrected ages is the projection of standard deviation of L/T of 6 to12 aliquots of each sample into the constructed DRC.

7. DISCUSSION

The CP1 grossly underestimates the ages and yields only 20% compared to the expected ages. In our samples, even first and second assumptions are satisfied third assumption would not have been satisfied. Third assumption will be invalid if the sample is too old and the fading rates are high (Huntley and Lamothe, 2001). If the sample is too old and for higher fading rates, the fading scheme can not be approximated with the logarithmic decay for the whole burial period. Hence the fading correction by extrapolating the fading of laboratory induced signals to the burial period in million years would give, most probably, an underestimated age. The failure of third assumption could be the reason for the huge underestimation of ages by CP1.

The CP2 corrected ages were also underestimated and yielded only 40% compared to the expected ages. Assumed values for s, α and barrier height are $3 \cdot 10^{15}$ s^{-1}, $9 \cdot 10^{9}$ m^{-1} and 3 eV respectively and they are related to OSL_{IR-B} signal from feldspar. For the OSL_{B-UV} [360ºC] signal, used in this study, the s value is not available. If we change s value to 10^5 s^{-1} also remove the factor 1.8

from the fading equation (**Eq. 6.2**), then the obtained arithmetic mean of ρ' is $(26.5 \pm 0.4) \cdot 10^{-6}$ (range: $21.0 \cdot 10^{-6}$ to $33.2 \cdot 10^{-6}$). If one reconstructs natural DRC using maximum value of ρ $33.2 \cdot 10^{-6}$ then the corrected ages are consistent with the expected ages for 5 out of 8 samples (**Table 4**). The constructed DRCs using both the s ($3 \cdot 10^{15}$ and 10^5 s^{-1}) are given in **Fig. 9**. The corresponding nearest neighbour luminescence centre distribution at various times in nature are plotted in **Fig. 10** for a) $s = 3 \cdot 10^{15}$ and b) 10^5 s^{-1} respectively.

It can be noticed that only a small fraction of the signals is stable using which the natural DRC's are constructed (**Fig. 9**). The regenerated signals constructed using the latter value is more stable as compared to the former. This might partly account for the age underestimation. These ages are encouraging and require further investigations.

8. SUMMARY

OSL_{IR-B} [50ºC], OSL_{IR-B} [200ºC], OSL_{B-UV} [50ºC] and OSL_{B-UV} [225ºC] are found that these signals can not be used to estimate the ages for the volcanic samples studied
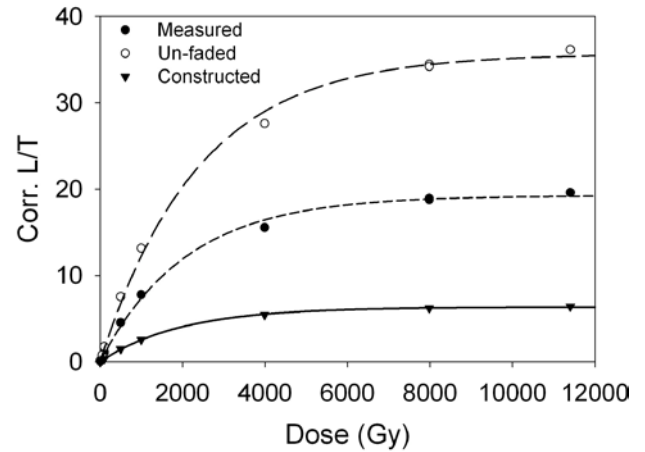

Fig. 8. *The measured dose response curve, unfaded dose response curve and the constructed natural dose response curves are shown. The highest natural L/T point is also shown.*

Table 4. *Comparison of corrected ages by CP2 for two values of attempt-to-escape frequency factor, s. '-' sign indicates overestimation.*

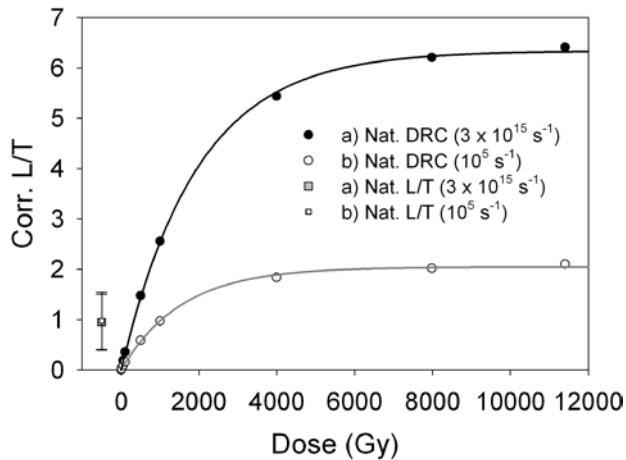| Sample	Corrected age$_{CP2}$ for s = $3 \cdot 10^{15}$ s^{-1} (ka)	Corrected age$_{CP2}$ for s = 10^5 s^{-1} (ka)	Expected age (ka)	Under or over estimation (%)
FL2	407±22	1139±140	1000-1500	-2
FL3	168±15	305±32	400	15
FL4	350±57	835±248	2000-2200	48
FL5	193±6	360±13	500-670	34
FL6	157±46	324±116	400	-10
FL10	362±33	856±138	670	-7
FA2	124±8	228±16	< 1000	51
FA5	5±1	8±2	< 1000	98

Fig. 9. *The constructed natural DRCs using attempt-to-escape frequency factor, s, a) $3 \cdot 10^{15}$ s^{-1} and b) 10^5 s^{-1}. For clarity the natural L/T points are plotted earlier in the Dose axis.*
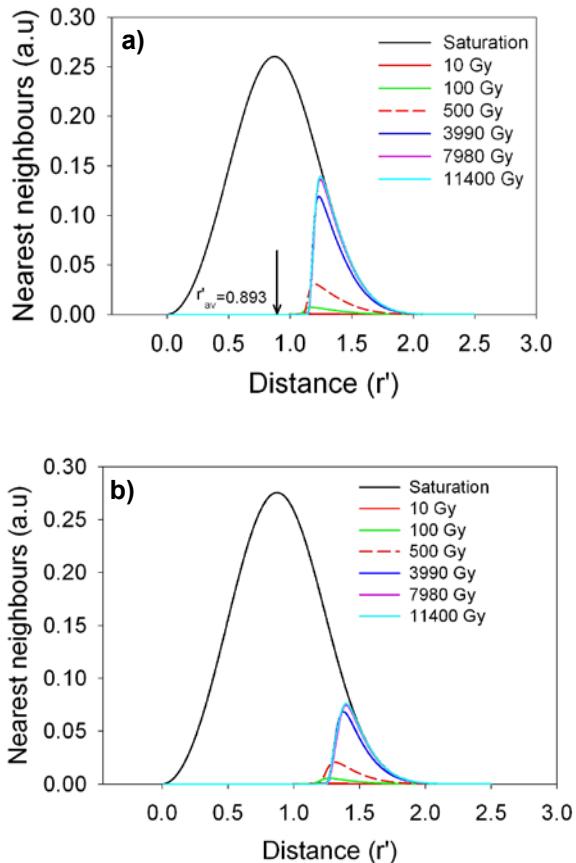

Fig. 10. *The nearest neighbour hole distribution at various times (doses for a constant dose rate) in nature obtained using s, a) $3 \cdot 10^{15}$ s^{-1} and b) 10^5 s^{-1}. The abscissa is the dimensionless distance variable, r' as given in Eq. 6.3. Stable signals commences from r' a) ~ 1.03 and b) ~ 1.20 and r'_{av} is indicated for comparison.*

here. OSL_{B-UV} [360ºC] signal was used to date the volcanic eruption event of 8 basaltic rock samples. The measured ages were underestimated compared to the expected ages and the underestimation was attributed to anomalous fading. We applied two procedures 1) Huntley and Lamothe, 2001, and 2) Kars *et al.*, 2008 for fading correction. Using the latter correction procedure, the corrected ages have a deviation of more than 60% from the expected ages. However, if the *s* value is changed from $3 \cdot 10^{15}$ s^{-1} to 10^5 s^{-1}, then the corrected ages have a better agreement with the expected ages. This is an interesting result worth further investigations although we acknowledge that our *s* value is unrealistically small.

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APPENDIX

The nearest neighbour distribution function, $p(r')$ is shown as below (Huntley, 2006),

$$p(r')dr' = 3r'^2 \, e^{-r'^3} \, dr' \qquad (A.1)$$

where

$$r' = \left(\frac{4\pi\rho}{3}\right)^{1/3} r \qquad (A.2)$$

and ρ is the number density of luminescence centers (m^{-3}). And ρ itself is related to ρ' via

$$\rho' = \left(\frac{4\pi}{3\alpha^3}\right)\rho \qquad (A.3)$$

where α is assumed to be $9 \cdot 10^9$ m^{-1} and this corresponds to barrier height of 3 eV.

The average r' is calculated as below (Chandrasekhar, 1943),

$$r'_{av} = \int_0^\infty r' \, p(r') dr' \qquad (A.4)$$

Taking $t = r'^3$; $dt = 3 \, r'^2 \, dr'$, and after substituting these in **Eq. A.4**,

$$r'_{av} = \int_0^\infty t \, e^{-t} \frac{dt}{t^{2/3}} = \Gamma\left(\frac{4}{3}\right) = 0.893 \qquad (A.5)$$

The r'_{max} is calculated by equating the first derivative of $p(r')$ to zero as below,

$$\frac{dp(r')}{dr'} = r' e^{-r'^3} \left(6 - 9r'^3\right) = 0 \qquad (A.6)$$

$$r'_{max} = \left(\frac{2}{3}\right)^{1/3} = 0.874 \, . \qquad (A.7)$$

Using **Eqs. A.2** and **A.5**, r_{av} is calculated as 0.55407 $\rho^{-1/3}$ and r_{max} is calculated to be $0.54202 \, \rho^{-1/3}$ using **Eq. A.2** and **A.7**. For a given r value, the life time can be calculated using **Eq. A.8**,

$$\tau_{av} = s^{-1} e^{\alpha r_{av}} \qquad (A.8)$$

For example, $\rho' = 3 \cdot 10^{-6}$, $r_{av} = 6.88$ nm and the corresponding average life time is 8.21 ka. For $\rho' = 4.94 \cdot 10^{-6}$, $r_{av} = 5.93$ nm and the corresponding average life time is 0.625 years.