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Efficacy of fading correction procedures to date Late Quaternary Higher Central Himalayan lacustrine sediment

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Abstract: **Feldspar luminescence dating method has a wider dynamic dating range than quartz due to its larger sensitivity and higher luminescence saturation level. However, feldspar luminescence fades 'anomalously' with time that generally underestimates the age using feldspar. To correct the anomalous fading of the luminescence signal, three procedures have been devised. We examined the efficacy of fading correction procedures on lacustrine sediment samples from the Central Himalayan region. We observed that two correction procedures (HL and LM) are insufficiently corrective, but HK works well.**

Index Terms: **Anomalous Fading, Fading Correction, Feldspar IRSL, Kunti Banar Basin, Kalla Glacier, and Luminescence Dating.**

I. INTRODUCTION

Luminescence dating technique provides reliable depositional ages for the Late Quaternary sediments (Fuchs & Owen, 2008; Rhodes, 2011; Wallinga, 2008). This method widely uses quartz from the sedimentary material and can date materials typically of 100 kyr old. This limitation is imposed particularly by the saturation of luminescence signals in quartz. That means the older sedimentary quartz grains have no available traps to keep the electrons, because all the traps have been filled. However, since feldspar has more impurities than quartz, it holds more trapped electrons. As a result, feldspar may be used to date sediments older than 100 kyr. When we mention feldspar, we are referring to K-feldspar, which has well-established methodology for luminescence dating compared to Na- and Ca-feldspar. Similarly, feldspar luminescence refers to infra-red stimulated luminescence, or IRSL. Although feldspar has advantage in this regard, its luminescence anomalously fades with time and it is temperature independent (Spooner, 1994). This anomalous fading of the feldspar luminescence leads to the underestimation of ages of the sedimentary material using feldspar.

Since the anomalous fading of luminescence in feldspar was reported, there are studies to understand and circumvent the issue. As a result there are three fading correction procedures to correct for the fading. In this study, we want to see how well the three fading correction procedures work in terms of providing consistent age estimations. If the fading correction procedures do not provide consistent ages, reason(s) may be sought for the inconsistency. Although there are a few studies that have tested the performance of these fading correction procedures, they mainly focus on the saturated and older samples (King, et al., 2018 & Li et al., 2018). Here we focus on the samples that were 1.5 - 30 kyr old. The objectives of this study are,

1. to compare the feldspar luminescence ages corrected for anomalous fading using three correction procedures, and
2. to determine which approach is better in a particular context.

II. SAMPLE DETAILS

To achieve the aforementioned goals, we used 12 samples that have previously been dated using luminescence dating technique for palaeoclimate reconstruction in the Central Himalaya (Singh et al. in Preparation). These 12 sediment samples were taken at various depths from a 4 m excavated trench in the center of a relict lake in the Kunti Banar river basin of the Kalla glaciated valley from Higher Central Himalayan region. These samples have been dated using IRSL signal from the extracted polymineral fine grains (4-11 μm). Since, the quartz grains were significantly contaminated by IRSL emitting micro-inclusions, and hence dating them was not straightforward (Singh et al.,

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Under Review). Apart from unavailability of pure quartz grains, fine grains were preferred because they might have been well bleached during transportation, as they tend to stay in water suspension for a relatively longer period and hence get enough Sun exposure. The fading-corrected ages were used to establish the chrono-stratigraphy of the lake profile under study. We used a fading correction procedure that was theorized by Huntley (2006) and implemented by Kars et al. (2008). The finalized (fading-corrected) ages for this relict lake profile ranged from 1.5 (± 0.1) kyr to 14.6 (± 1.1) kyr.

III. ANOMALOUS FADING OF FELDSPAR LUMINESCENCE

The phenomenon of anomalous fading was first reported on thermoluminescence of the lunar samples (Wintle, 1973; Wintle, 1977). It was unusual (anomalous) because the fading of luminescence could not be explained by the known thermal kinetics, and in fact the fading appeared to be independent of the sample storage temperature (Spooner, 1994). Since then efforts are being made to understand this anomalous phenomenon. The anomalous fading was gradually understood to be nothing more than phosphorescence (after-glow) emitted during tunneling assisted recombination of electron-hole (donor-acceptor) pairs without any involvement of the conduction band, as in the case of quartz (Visocekas et al., 1976; Visocekas, 2002). This after-glow follow a power law decay. This power law behavior of after-glow/phosphorescence results in a logarithmic decay (Huntley & Lamothe, 2001) or exponential decay (Huntley, 2006) of the concentration of trapped electrons, if the finite irradiation time is considered in the calculation (Delbecq et al., 1974). To address this, fading correction is performed, which consists of two steps: 1) measuring the fading rate and 2) implementing the correction, both of which are reliant on the respective theoretical foundation used to explain the anomalous fading phenomenon.

A. *Measurement of anomalous fading rate*

Fading rate is measured using multiple sensitivity corrected IRSL (L/T) measurements with varying time delays between the cessation of irradiation and IRSL measurements (L) following preheat (Auclair et al., 2003). Any sensitivity change in the sample is corrected by the test dose luminescence.

1) *Logarithmic decay (g-value; %/decade)*

As demonstrated in Eq. 1, a plot of sensitivity corrected IRSL (L/T) versus ln(t*) and its linear fit yields g-value (= 100. -slope/intercept). The time since irradiation is denoted by t*, which is computed as the sum of half of the irradiation time, preheating and holding time, and the time until the IR LEDs are shining on the sample. The g-value that was measured thus using t_c=1 hour is standardized as g-value$_{tc=2days}$ for t_c=48 hours.

$$\frac{L}{T} = \left(\frac{L}{T}\right)_0 \left[1 - \frac{g}{100} \times \log\left(\frac{t^*}{t_c}\right)\right] \qquad (1)$$

where L/T and $(L/T)_0$ are the sensitivity corrected IRSL at different delay times and at t_c, respectively. This function (Eq. 1) predict a zero and negative L/T at a larger t*, or even smaller t* if g-value is large. This may be physically meaningless but the mathematics allows that to happen and this is the limitation. The fading behavior of KBPL 07 is shown in Fig. 1 and the data were fit to Eq. 1 (hyphened line). The fading parameter, g-value$_{tc=2days}$ of KBPL 07 is 1.1 (± 0.5) %/decade and the fading parameter for all the other samples are given in Table I. They range from 1.1 to 2.3 %/decade.

2) *Exponential decay (ρ')*

The same set of measured data is used to calculate the ρ′ by fitting the data to the equation Eq. 2 as given below,

$$\frac{L}{T} = \left(\frac{L}{T}\right)_0 e^{-\rho'[\ln(1.8\times s\times t^*)]^3} \qquad (2)$$

where s is the attempt to escape frequency (3×10^{15} s^{-1} and t* is the time since irradiation, L/T and $(L/T)_0$ are the sensitivity corrected IRSL at different delay times and at nearly 0 s respectively. The ρ′ is the measure of number density of electron-hole pairs inside an irradiated feldspar grain. According to this function (Eq. 2), near zero fading rate is observed both at the smaller delay times and larger delay times. It is to be noted that this function will never allow the L/T to be zero as Eq. 1 does. So in this aspect Eq. 2 is better describing the anomalous fading than do Eq. 1. The fading behavior of KBPL 07 was fit to Eq. 2 as well and shown in Fig. 1 (thick line). A general description of fading behavior is Eq. 2 whereas Eq. 1 approximately describes. The fading parameter, ρ′, of KBPL 07 is 0.9 (± 0.3) x 10^{-6} and all the fading parameters are given in Table I. They range from 0.9 x 10^{-6} to 1.7 x 10^{-6}.

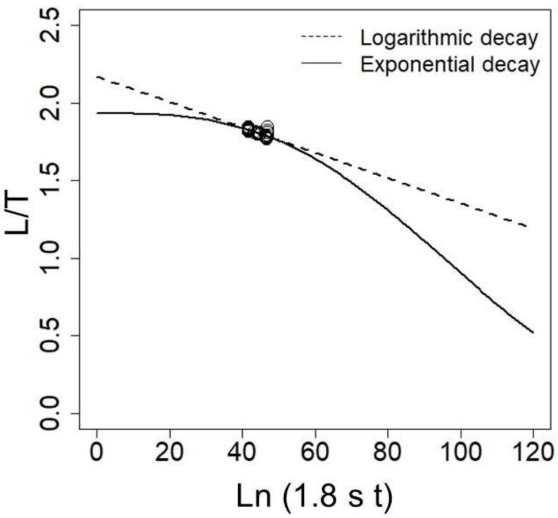

Fig.1. Fading rate parameters obtained by fitting to logarithmic (Eq. 1) and exponential (Eq. 2) decay functions (KBPL 07)

B. Procedures to correct for anomalous fading

There are, in fact, four correction procedures. The first is called HL and is based on Huntley and Lamothe (2001). The second, which we name LM, is based on Lamothe et al. (2003). The third is based on Wallinga et al. (2007). As this correction (3^{rd}) and LM provide similar outcomes (Li et al, 2019), and LM is the foundation of the third procedure, we focus solely on LM in our research. These three procedures are based on the logarithmic fading behavior and use g-value as the fading parameter in their correction procedures. The fourth procedure is based on Huntley (2006) and was implemented by Kars et al. (2008) and we call it HK. This is based on the exponential fading behavior and ρ' is used as the fading parameter in the correction procedure.

1) HL Procedure

This is the most widely used fading correction procedure. As mentioned earlier, this procedure is based on the logarithmic fading behavior (Eq. 1). The fading correction is done by solving Eq. 3 by iteration,

$$\frac{T_f}{T} = \frac{De_f}{De} = \frac{L/T}{L/T_0} = 1 - k\left[ln\left(\frac{T}{T_c}\right) - 1\right] \qquad (3)$$

where the parameters with and without '*f*' subscript are faded age, D_e and L/T and fading corrected ones, respectively. The g (g-value) is related to k using this relationship, **g = 100 . k . ln10**. The T_c is the characteristic time and is equivalent to the time between sample collection and measurement. As Eq. 3 suggests this procedure will work as long as the linear relationship is maintained between the sensitivity corrected IRSL (L/T), D_e and age (Morthekai et al. 2008, 2011). As this linearity is expected for the samples of older up to ~20 kyr, this method can only perform for those samples that are younger than 20 kyr. The *calc_FadingCorr()* method as implemented in ***Luminescence*** package (Kruetzer et al., 2021) in R platform (R Core Team, 2021) was used to compute this.

2) LM Procedure

As the previous procedure explicitly states its limitation i.e., HL will only work as long the natural luminescence falls in the linear part of the growth curve (also known as dose response curve), this method claims to work even in non-linear part of the growth curve. This procedure corrects the natural L/T using the g-value and natural dose rate before interpolating onto the measured growth curve (Fig. 2). The *calc_Lamothe2003()* method was used to perform this. We used single saturating exponential function to describe the growth curve except for 3 samples (KBPL 05-07) where general order kinetics was used. Similarly the values of all the measured aliquots were fading corrected. Then the central age model (CAM) was employed to calculate the representative D_e for age calculation (Galbraith et al., 1999). Fading corrected ages are given in Table I.

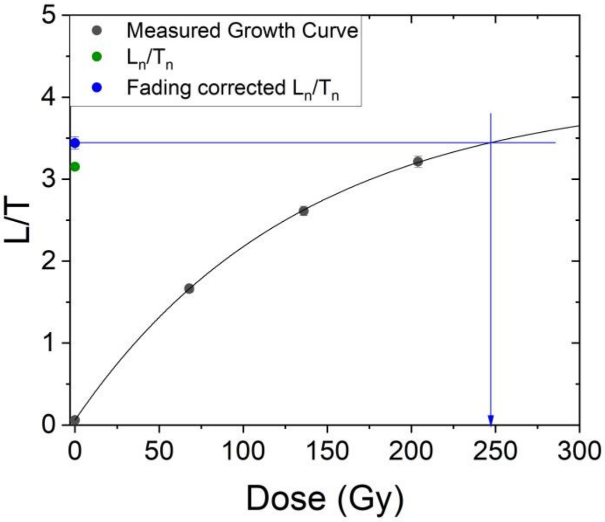

Fig.2. Fading correction by LM procedure (KBPL 07)

3) HK Procedure

This method also claims to work throughout growth curve. The LM corrects the natural L/T, whereas this method correct the measured growth curve to construct the unfaded growth curve before simulating the naturally faded growth curve from the unfaded growth curve (Morthekai et al., 2011; Biswas et al., 2013). Then the measured natural L/T was interpolated onto the natural growth to get the fading corrected D_e (Fig. 3). The *calc_Huntley2006()* method was used to compute this. As we used for LM, general order kinetics was used for three samples (KBPL 05-07) and single saturating exponential function to describe the growth curve for other samples. The D_e's of all the measured aliquots were fading corrected and the CAM was employed to calculate the representative D_e for age calculation. Thus fading corrected ages are given in Table I.

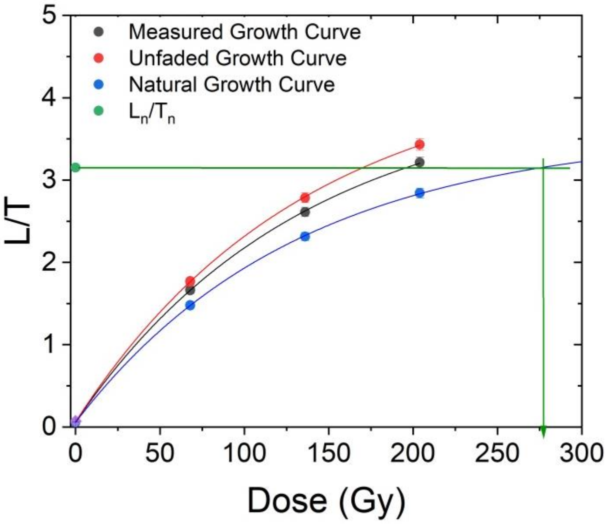

Fig.3. Fading correction by HK procedure (KBPL 07)

Table 1: Fading parameters (g-value and ρ′) and the fading corrected IRSL ages are given against depth.

Sample Code	**Depth (cm)**	**g-value$_{tc=2days}$ (%/decade)**	**ρ′ $(\times 10^{-6})$**	**HL (kyr)**	**LM (kyr)**	**HK (kyr)**
KBPL 12	25	2.3 ± 0.5	1.7 ± 0.3	1.0 ± 0.1	1.3 ± 0.1	1.5 ± 0.1
KBPL 11	55	2.2 ± 0.4	1.5 ± 0.1	4.5 ± 0.2	5.7 ± 0.1	7.2 ± 0.6
KBPL 10	80	2.4 ± 0.5	1.5 ± 0.5	2.1 ± 0.1	2.6 ± 0.1	2.8 ± 0.1
KBPL 09	110	2.2 ± 0.4	1.6 ± 0.2	3.3 ± 0.2	4.2 ± 0.1	4.4 ± 0.2
KBPL 08	140	1.8 ± 0.5	1.5 ± 0.5	4.4 ± 0.2	5.3 ± 0.2	5.7 ± 0.2
KBPL 07	180	1.1 ± 0.5	0.9 ± 0.3	15.5 ± 0.8	19.3 ± 1.2	25.7 ± 2.4
KBPL 06	205	1.6 ± 0.4	1.1 ± 0.4	11.4 ± 0.9	14.6 ± 0.8	17.6 ± 0.9
KBPL 05	235	1.9 ± 0.5	1.4 ± 0.3	13.2 ± 1.6	19.8 ± 1.5	29.3 ± 0.3
KBPL 04	265	1.2 ± 0.4	0.9 ± 0.3	6.9 ± 0.6	8.8 ± 0.5	9.3 ± 0.5
KBPL 03	300	1.4 ± 0.5	1.1 ± 0.3	9.0 ± 0.6	10.9 ± 0.5	11.7 ± 0.6
KBPL 02	340	1.9 ± 0.3	1.3 ± 0.4	5.6 ± 0.4	7.6 ± 0.7	7.5 ± 0.3
KBPL 01	370	3.2 ± 0.4	2.2 ± 0.2	10.1 ± 1.2	12.2 ± 0.6	14.6 ± 1.1

IV. DISCUSSION

All the fading corrected ages using the three fading correction procedures are given in Table I. There are four samples (KBPL 05-07 and 11) that showed poor bleaching characteristics and hence they were expected to be stratigraphically inconsistent (Singh et al., in Preparation). However for the purpose of testing the fading correction procedures, these out of tune samples (KBPL 5-07 and 11) may be included to see the consistency among the three sets of fading corrected ages.

So rather than comparing the ages in an age-depth plot, it would be much useful to compare them on a scatter plot, as shown in Fig. 4. Both HL ages and LM ages are compared with HK ages that are in the x-axis. For better visualization there is drawn a 1:1 line with 10 % error (grey shaded region). Almost all the HL ages lie outside (below) the grey shaded area and hence underestimated more than 10 % of HK ages. Among the 12 LM ages, 4 ages lie below the grey shaded area.

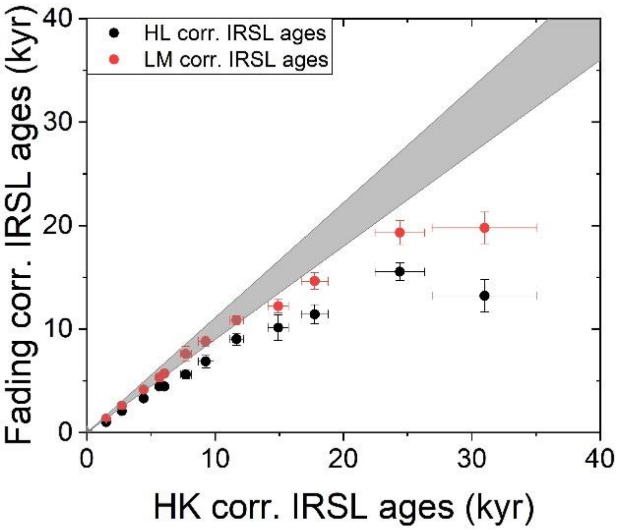

Fig. 4. Comparison of fading corrected ages among each other.

The HL procedure informs us that this method work well for the samples that are younger than 20 kyr. In this profile, only 3 samples (KBPL 05-07) are older than 20 kyr. So except these 3 samples, HL procedure should have corrected well but it did not do. The fading rate values are also not higher (more than 10 %/decade) and hence that is not the reason for HL's underestimated ages. The reason must be the natural L/T lie in the non-linear part of the growth curve. Also the reason why IRSL signal is almost on the saturation even in the younger samples is the higher dose rate (max. dose rate 30 (± 1.3) Gy.ka^{-1} for KBPL 02; Singh et al. in Preparation). Had the dose rate values normal, certainly 9 samples (except 3 samples older than

20 kyr) would have been fading corrected within 10 % as HK procedure.

Regardless of LM's claim that it can correct effectively well even when the natural L/T lie on the non-linear part of the growth curve, four samples conspicuously deviated (underestimated) by more than 10% from HK ages. This is contrast to what was observed (Li et al., 2018). In order to explain why these 4 values were underestimated, we extracted the normalized natural IRSL (n/N) and saturated IRSL (n/N_{SS}) data that were computed during HK fading correction in *Luminescence* package. The n/N was calculated from the measured growth curve and n/N_{SS} was calculated from the simulated natural growth curve. The n/N_{SS} is expected to be similar among the samples and not more than unity. The n/N is expected to increase with age of the samples. There are four samples that had similar n/N and n/N_{SS} values (Fig. 5). The red rectangle indicates n/N_{SS} values with 2 σ. It is observed that if n/N is similar to n/N_{SS}, then LM cannot correct for fading.

Regarding HK's performance, it is difficult to ascertain that HK fading corrected ages are the correct ages, in the absence of independent chronology. However, although not independent, two luminescence ages (4 ± 2 kyr for KBPL 09 & 24 ± 8 kyr KBPL 07) estimated using IRSL arising from feldspar micro-inclusions within quartz (Singh et al. Under Review) together suggests that HK has appropriately corrected. So, it can be stated that HK do correct for fading better than HL and LM.

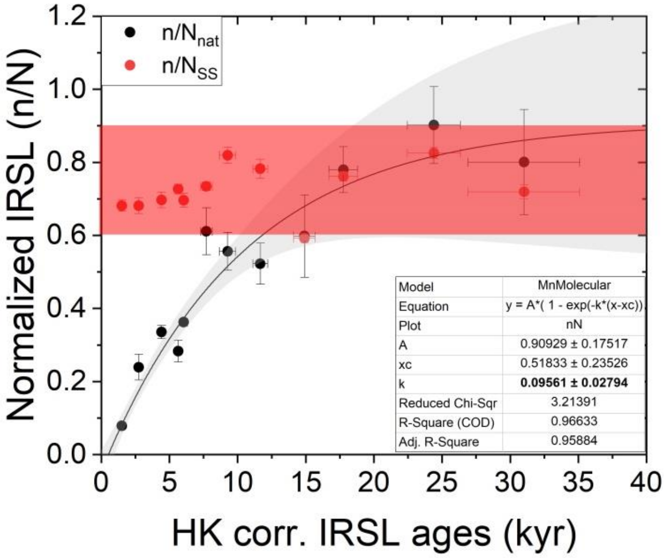

Fig. 5.Condition within which LM correction works.

CONCLUSION

Our findings show that there were inconsistent ages produced by the three fading correction procedures even for samples younger than 20 kyr. As higher dose rate would saturate the luminescence signal sooner, as is the case with our samples, HL procedure cannot perform well, although the fading rate values are normal. If n/N and n/N_{SS} are similar and comparable, then LM should not be used in such case. So only HK, do appear to have corrected the fading better here.

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