

## **Chapter-3**

### **Changes in natural OSL sensitivity: Implications to Dating**

#### **3.1 Introduction**

Single Aliquot Regeneration (SAR) protocol is used widely in almost all kind of sediments viz. aeolian, colluviums and water-lain. In recent times, development of SAR procedure has been paid attention in an exhaustive manner. A SAR measurement involves several steps of irradiation, preheat and luminescence measurement on an aliquot to estimate paleodose (Murray, et al., 1997; Murray and Roberts, 1997; Murray and Wintle, 2000; 2003). Preheat and luminescence measurement however can cause changes in the sensitivity (luminescence/absorbed dose-mass) of the sample. In a typical SAR measurement, this change is monitored and corrected for using an OSL output of a small test dose given after natural and regenerated OSL measurements. The robustness of such sensitivity changes is monitored again through a dose recycling point introduced in the SAR procedure.

It has been shown that 110°C TL peak was linearly correlated with OSL response (Stoneham and Stokes, 1991, Stokes, 1994a, 1994b). The 110°C TL peak's mean life is only few hours, which means that traps are unpopulated in a natural sample relating to 110°C temperature. Hence this peak can be used as a surrogate for sensitivity.

While incremental OSL and 110°C TL responses to test dose measured subsequent to the regenerative cycles of doses and OSL measurements are capable of fully quantifying sensitivity changes during construction of the regenerated growth curve, there remains some uncertainty as to whether the measurement of 110°C TL or OSL following stimulation of the natural OSL faithfully describes the behavior of the sample prior to stimulation and preheating. The studies have shown that the most serious sensitivity changes occurred as a result of the erasure of the natural signal (Stoneham and Stokes, 1991; Stokes 1994a, 1994b). Such changes can lead to systematic offset from true paleodoses depending upon the luminescence behavior. Present work examined sensitivity change of a sample during natural OSL measurement in a SAR procedure on quartz extract from Himalayan sediments and suggests the possible corrections to minimize the effect of such sensitivity changes, however, focus of the study is to report the sensitivity changes and its effect on age estimation.

In the present study, the sensitivity of the 110°C TL peak was measured of a small test dose before and after OSL measurement to monitor any sensitivity changes that would have occurred during OSL measurement. Changes in sensitivity during the preheat and measurement of natural OSL have not been considered in a SAR protocol and this could imply systematic offsets in ages (Stokes and Singhvi, under preparation). In view of that, these authors have examined some samples from various archives and have shown that initial (natural) sensitivity varied significantly. The change in sensitivity ranged from an increase by up to 27% and a decrease by up to 30%. A natural sensitivity corrected SAR (NSC-SAR) procedure was proposed by these authors. NSC-SAR protocol has been discussed in detail in chapter-2 of the thesis), which monitors any sensitivity change during natural OSL measurement and in the process makes a SAR protocol, truly robust.

### **3.2 Experimental details and methods involved**

The Quartz fraction from samples was extracted using the method discussed in detail in chapter 2. Small aliquots of 5mm diameter were used for analysis. For constructing growth curve, initial 2 seconds (from 100 seconds of OSL measurement) of a typical shine down curve was taken with a background subtraction from the same curve.

In present method, additional steps of test dose and its 110°C TL peak measurement was done apart from standard SAR measurement. The first test dose measurement ( $T_1$ ) represents the sensitivity of natural sample prior to any OSL measurement. Measurement ( $T_4$ ) represent the sensitivity of the sample after its preheat and OSL measurement. In present method a term “natural correction factor” (ncf) has been introduced (Stokes and Singhvi, under preparation) which has been defined as the ratio of  $T_1$  and  $T_4$ . Ideally  $ncf=1$ , if there is no change in sensitivity and  $ncf>1$  indicates drop in the sensitivity and vice versa.

In the standard SAR method, a growth curve is constructed by giving laboratory beta doses to the aliquot followed by preheat and OSL measurement (Murray & Wintle 2000). The luminescence yield at each dose point is corrected for sensitivity change by measuring the OSL yield for a test dose. A plot of the sensitivity corrected regenerated OSL signal with dose enables the construction of a OSL-dose growth curve and sensitivity corrected natural luminescence intensity is interpolated onto this curve to obtain the equivalent dose,  $P$ . In this procedure, natural signal is corrected using test dose signal and ncf for any sensitivity changes and then it is read out on regenerated OSL growth curve (Fig. 2.7 and 2.8, chapter 2). We have compared the Paleodoses separately to see its effect on the minimum 10% and average of a dose distribution obtained after running SAR.

In this method, the ratio of two TL signals is used to correct the sensitivity changes occurred in the OSL output of the natural signal. The corrections would be applicable only if (i) the TL signals correlates linearly with the OSL signal and (ii) the intercept of the line of correlation is zero. Correlation of the TL and OSL signal was examined by using a few aliquots were taken from some samples and cleaned for any natural signal present in it using blue light stimulation. After that, each disk was given a series of beta doses (ranging from 1.2 – 6Gy) followed by a TL measurement (temp. up to 160°C) followed by OSL measurement. A graph was plotted for each disk for TL against OSL (Fig. 3.1).

The method was applied to several samples of different parts in Himalaya. For this samples from various locations were collected. These are discussed in detail in Chapter 5 of the thesis.

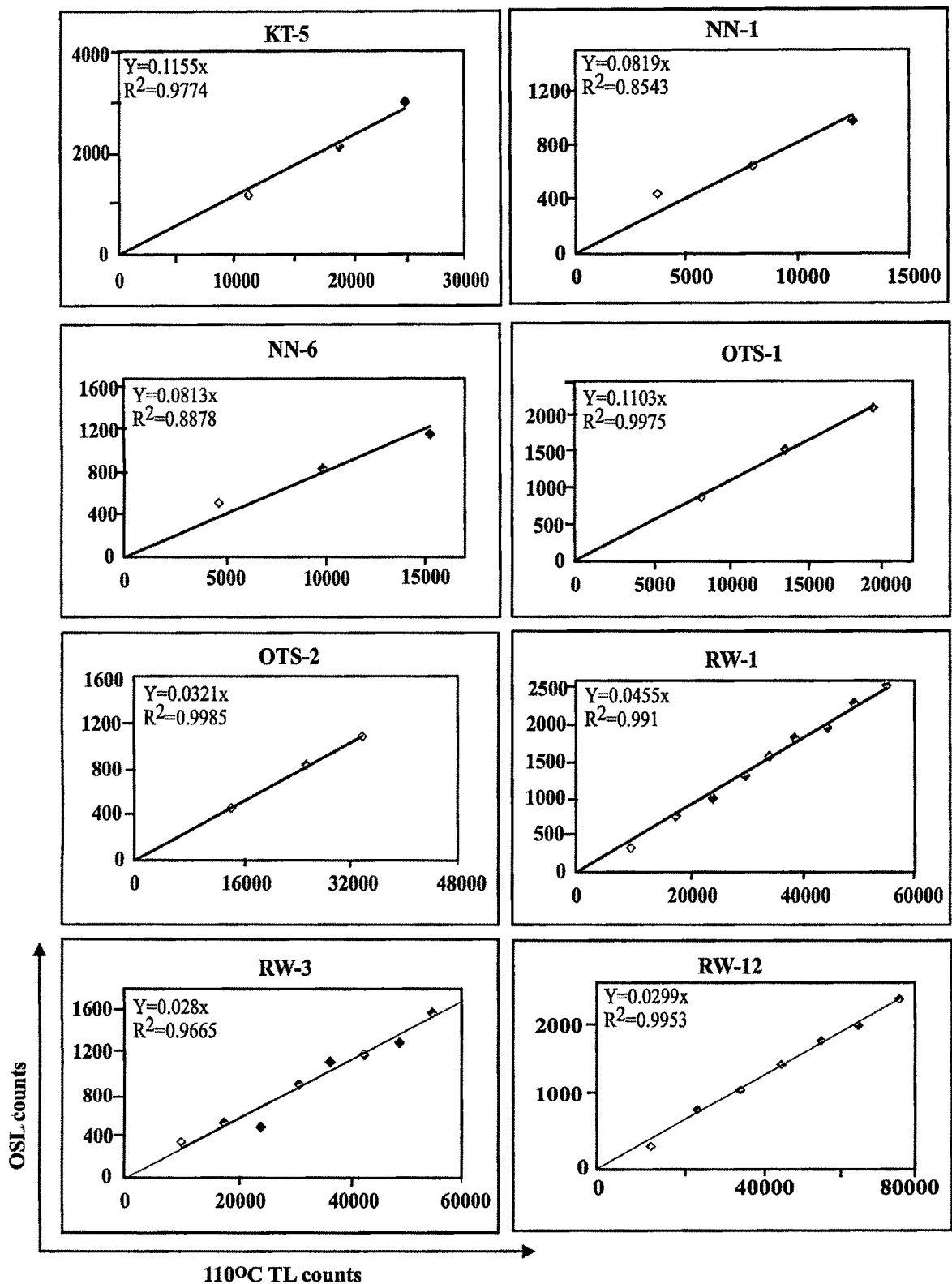


Figure 3.1. Correlation of 110°C TL counts and OSL counts. Dose points starts with 1.2 Gy and rest are in multiple of that. (Regression line has been forced to pass through origin)

### 3.3 Results and Discussions

Total 14 samples were examined. It was observed that the regression line of TL and OSL is showing a good correlation having high values of regression coefficient ( $R^2 > 0.85$ ) (Fig. 3.1). These lines were forced to pass through origin considering that in ideal cases TL and OSL should be equal for zero doses. The sensitivity was checked in the new protocol to observe the effect of both of preheat and OSL separately. To observe this, ratio of  $T_1$  and  $T_2$  was taken to represent any changes in sensitivity due to preheat only (Fig. 3.2). It is clear from the graph that the sensitivity changes is the cumulative effect of preheats as well as OSL measurement too. In the present study, the ncf was taken as the representative of cumulative effect of preheat and OSL measurement. The paleodose was computed with and without corrections and compared. In the extreme cases, ncf varied up to 50% however; typical ncf values ranged from 15 – 25%. The histograms (Fig. 3.3) show the corrected and uncorrected values of P's computed keeping the bin width identical. Gaussian curves were fitted for these distributions of doses and the full width at half maximum (FWHM) of the curve was computed to observe any effect after applying ncf correction. It is clear from the few graphs that FWHM of the Gaussian curve decreased from 20 – 40% after corrections for natural sensitivity changes. It is clear from the histograms and the Gaussian curves that the paleodose distribution improved after ncf sensitivity correction. The average of ncf corrected Paleodoses indicated a significant offset (~7 – 38%) in the ages from the uncorrected ages using the conventional SAR protocol (Table 3.1). As seen from a significant reduction in the FWHM of Gaussian curve of paleodose distribution after ncf corrections, it is evident that sensitivity changes is responsible in some samples for dose heterogeneity which would have been attributed to partial bleaching. This is important in partially bleached sediments of a young sample (1-2 ka) where least values or minimum age model is preferred (Jain et al, 2004). Hence it is recommended that this correction should be used for SAR paleodose estimation.

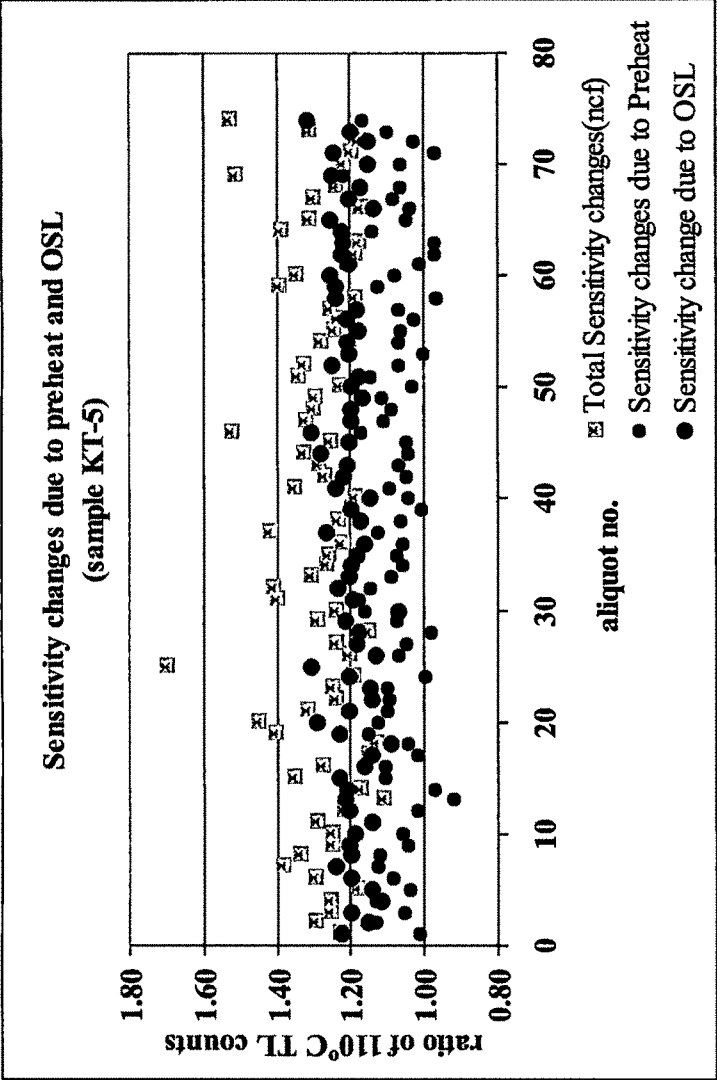


Figure 3.2. Sensitivity changes due to preheat and OSL stimulation alone.

Table 3.1. Effect of natural sensitivity changes on the average paleodose. % Change has been calculated by taking ratio of difference between the corrected and uncorrected paleodose to the uncorrected paleodose.

Sample	Paleodose (average)(Gy)		% Change
	Without sensitivity correction	After sensitivity correction	
NN-1	46 ± 11	37.6 ± 11	18
NN-5	29 ± 6	25 ± 6	14
NN-6	25.7 ± 7.3	23.4 ± 7.7	9
KT-5	11 ± 4	7 ± 4	36
KT-6	8 ± 3	6 ± 3	25
OTS-1	30.4 ± 9.2	28.2 ± 9.7	7
OTS-2	5.7 ± 1.6	4.9 ± 1.5	14
OTS-3	27 ± 7	21.9 ± 5.5	18
OTS-4	27 ± 14	19.8 ± 9.2	26
OTS-5	2.3 ± 0.6	2.1 ± 0.5	9
RW-1	22.8 ± 9.8	18.7 ± 8.5	18
RW-2	7.8 ± 4	6.3 ± 2.6	10
RW-3	12 ± 5	9 ± 4	19
RW-4	28.5 ± 11	22.5 ± 8	21

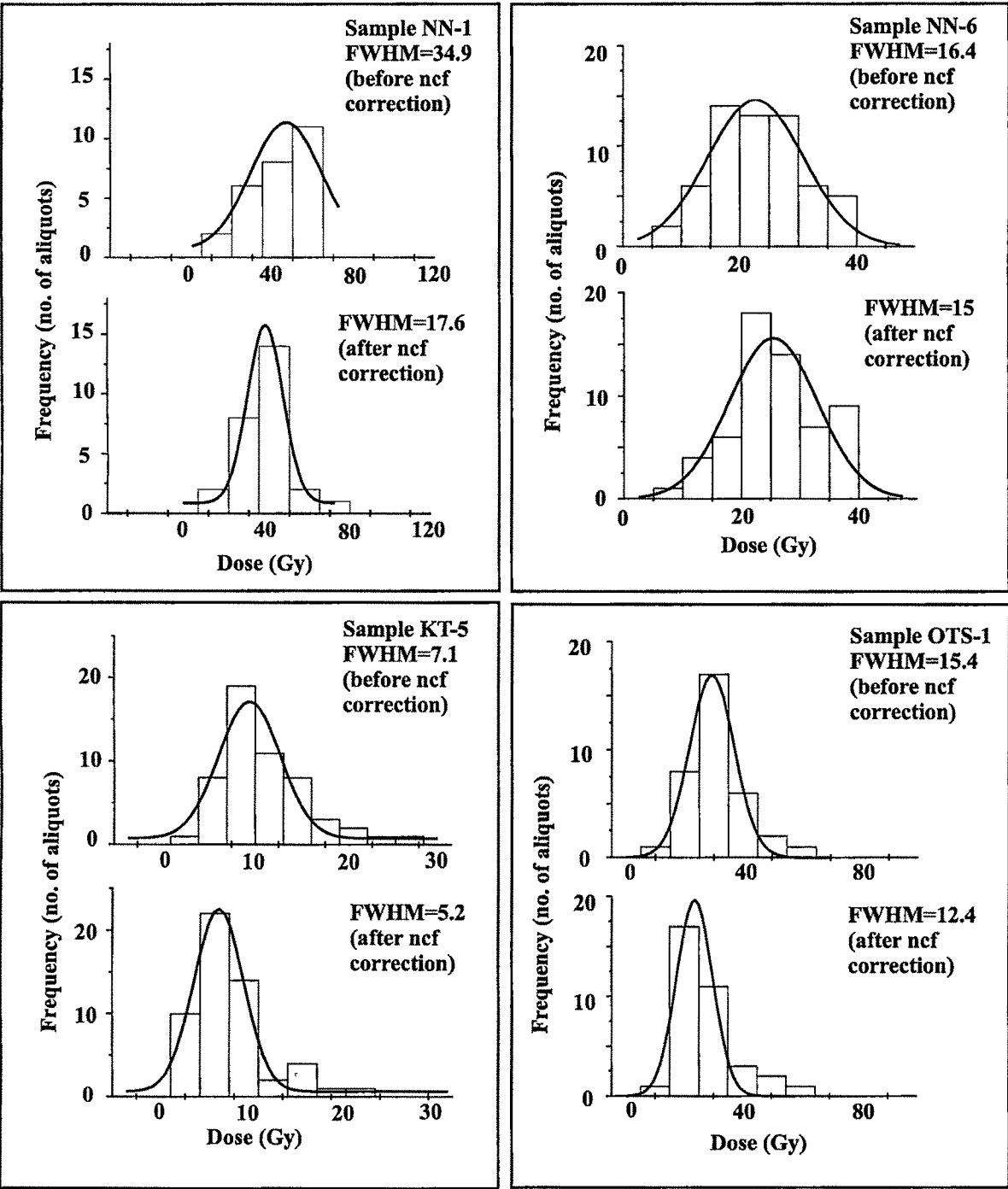


Figure 3.3. The effect of ncf correction on the shape of the dose distribution curve is seen by the decreased FWHM of the curve after correction.



## References

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