CHAPTER 6

OSL GEOCHRONOLOGY

Introduction

There are many dating methods which are used to calculate the time of formation of organic and inorganic materials, in which radiocarbon dating is the more popular. However, to get direct age of the time of deposition of sediments only few methods are available. Luminescence dating methods are the major among them for the Late Quaternary, wherein optical dating is proving to be progressively useful (Aitken, 1998). Luminescence dating is a chronological method and widely used in archaeological, anthropologic environments and many fields of earth and environmental sciences (Aitken 1998, Duller, 2008). It is based on the emission of light, means if we bombard the light source on to the substance / sediments, they will excite and emit energy. Some naturally occurring minerals such as quartz and feldspar have tendency to emit energy in the form of light, which is known as luminescence. This method is applied over an age range of less to over a hundred thousand years (Duller, 2008). In optically simulated luminescence (OSL) technique, the event which is the last exposure of the sediment to the sunlight. This exposure to sunlight is known as the bleaching event, which sets the remnant signal to near zero (Fig. 6.1). Later, when sediments get buried, the light is blocked by overlying sediments signal builds up again through the effect of exposure to the weak flux of ionizing radiation delivered by the radioactive elements such as ^{238}U-^{230}Th and ^{40}K in the sediment, as well as by cosmic rays and by rubidium-87 to a minor extent (Aitken, 1998).

In optical dating, the signal is stimulated by the optical luminescence and this signal is acquired by the exposure of the minerals from the sample to a beam of light source. This luminescence is measured by the highly sensitive device, which is known as Photomultiplier tube (Aitken, 1998). The acquired signal is usually called the natural OSL; this natural signal is compared with the artificial OSL regenerated signal using the laboratory irradiation. Then, this dating signal is compared with the signals obtained from the known doses of nuclear radiation, which have been

controlled from a calibrated radioisotope source; this allows calculation of the equivalent dose (D_e).

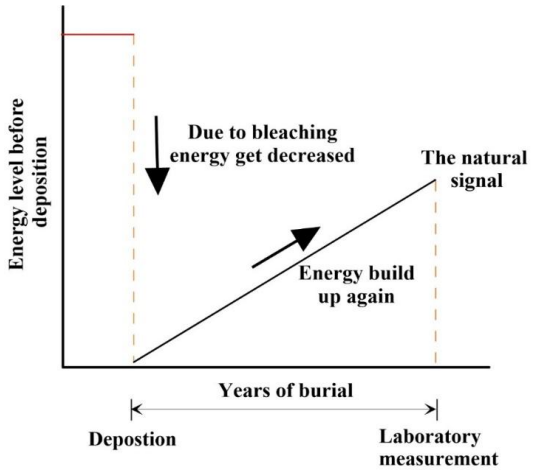

Fig.6.1 Shows bleaching event, subsequently the remnant signal builds up again through exposure to the weak natural flux of nuclear radiation.

Then the age is calculated by the following equation,

$$\textbf{Age (years)} = \frac{\textbf{Equivalent Dose } (D_e) \textbf{ (Gy)}}{\textbf{Dose rate (Gy/year)}}$$

The dose rate is the rate at which energy is absorbed from the flux of nuclear radiation and it is calculated by using the radioactivity of the sediment. Quartz and feldspar are used in optical dating method and use of zircon and volcanic glass has also been validated. Different stimulation wavelengths are used based on a choice of minerals and grain sizes. However, as a laboratory measurement protocol, namely the quartz OSL single aliquot regenerative dose (SAR) protocol of Murray and Wintle (2000, 2003) is used, because of its accurateness and reliability (Murray and Olley 2002, Rhodes et al. 2003, Duller, 2008).

Figure 6.2 presents a flowchart that is followed as a standard methodology in measuring the OSL geochronology. In the present study also the same has been followed and all measurements were

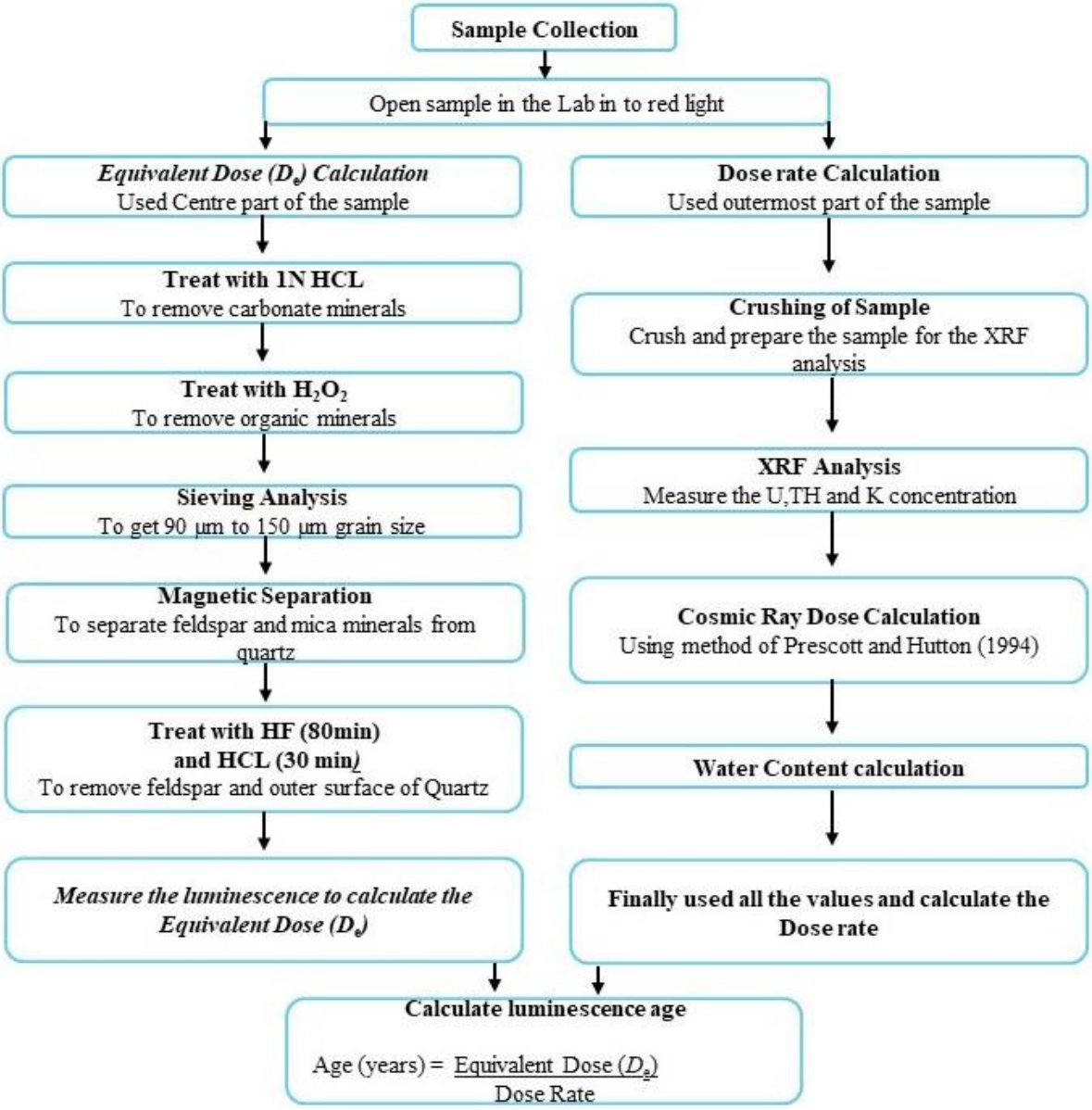

Fig. 6.2 Flowchart followed in OSL Dating in present study done at the Institute of seismological Research (ISR), Gandhinagar.

Application of OSL in dating Miliolites

Bhatt (2003) reviewed Saurashtra miliolite, which suggested that the basic difficulty with radiometric ages has been the diagenetic changes, later reworking of carbonates and their transport as grains by aeolian / fluvial agencies followed by cementation. This implies that a priori any age on bulk miliolite (Miliolidae grains + cement) will be younger than the age of individual grains of Miliolidae (cement being younger). In contrast, radiometric dating of the carbonate grains in deposits of aeolian and fluvial origin would provide overestimates, as the ages would correspond to the event of their formation and not the event of their aeolian or fluvial transport with eventual burial. Measurements of ^{230}Th / ^{234}U ages of miliolite samples ages in carbonates becomes unreliable due to interaction between grains and waters percolating through the sample during diagenesis. These alterations lead to unreliable ages (Gupta, 1991). In general, radiometric ages on bulk carbonates are inaccurate because of a complex sequence of events of deposition, cementation, and diagenesis (Sharma et al. 2017). Henceforth the present study employs optical dating of miliolite using detrital quartz grains only. As suggested by Sharma et al (2017) this chronometric application is based on the following some assumptions:

1. Quartz grains and allochems were synsedimentary.
2. The optically stimulated luminescence (OSL) of quartz grains was bleached to near zero during reworking as indicated by Rink (1999) and Murari et al. (2007).
3. The OSL of quartz grains was rebleached to near zero during reworking and aeolian transport to form carbonate dunes (Singhvi and Kar, 2004).
4. On burial, OSL reaccumulation began because of irradiation from ambient radioactivity, which continued unabated until excavation.

A distinctive advantage of OSL is that it dates the most recent event of deposition. Thus, for miliolite grains the OSL provides the age of their aeolian deposition and preservation.

OSL Methodology

The physical nature of samples dictated the manner of sample collection for OSL analysis. For consolidated rocks, large blocks were taken, and for friable samples, standard steel pipes were sed (Chandel et al., 2006). A total of 3 area sites and 4 OSL samples were analysed that included 3 chunk and one steel pipe sample.

An important aspect of the application of OSL was to ascertain if the carbonate coating on quartz grains was post depositional or that the grains had a carbonate coating at the time of transport and deposition. This is because the overall opacity (because of carbonate coatings) would have determined the ease of bleaching of the preexisting OSL signal in the quartz grains. The presence of micrite and sparite as cement in miliolite samples suggested that the carbonate coating was authigenic (i.e., post depositional). In thin sections the quartz grains show sharp contact with the microcrystalline cement, which also suggests that these grains did not have carbonate coatings prior to deposition. This inference accords with the results of Rink (1999) and Murari et al. (2007) and is supported by the experimental data on the distribution of doses by (Sharma et al. 2017). Extraction of quartz grains from the carbonate blocks was carried out under filtered red light. The outer 5-7cm of the blocks was removed by a stonecutter with water cooling. Similarly for the friable samples collected using stainless steel pipes, the two ends of the OSL pipes were removed and sample from the middle part of pipe was retrieved in subdued light conditions. The retrieved sample (from both cases) was treated with 1N HCl and H_2O_2 for removal of carbonates and organic matter. This was followed by separation of 90 to 150 micron fraction of sample using wet sieving method. The separated fraction was then dried and further subjected to magnetic separation using a Frintz Magnetic separator, from which magnetic and non-magnetic fractions minerals were separated. The non-magnetic fraction was then etched with 40% HF for 80 minutes followed by 12N HCl for 30 minutes. This removed the 25 ± 5 micron thick layer over the fresh quartz grain. The pure quartz grains hence extracted were washed with distilled water several times and then dried for the further analysis. The quartz grain were mounted on 10-mm stainless steel discs with silicon spray as adhesive for D_e measurements. The aliquots were made of 1 mm diameter. The luminescence measurements have been carried out in RISO, TL / OSL reader under blue LED source (470 ±30 nm). Beta irradiations were carried out using an on-plate ^{90}Sr / ^{90}Y beta source. Typically, about 15 to 40 aliquots per sample were measured for De. The choice of the model for the De calculation was based on over dispersion. The annual dose rate was computed using the elemental concentrations of potassium, thorium, and uranium and an adopted water content of 10%. The cosmic ray contribution to dose rate was computed using Prescott and Hutton (1994). The maximum water-holding capacity as estimated from thin sections was 8% to 12% of volume. Thus, the use of the 10% water content is justified. The elemental concentrations of U, Th, and K were measured using XEPOS HE X-Ray fluorescence

spectrometer. Figure 6.3 represents does estimation plots and the radial plots of the De values used for the calculation of the OSL age.

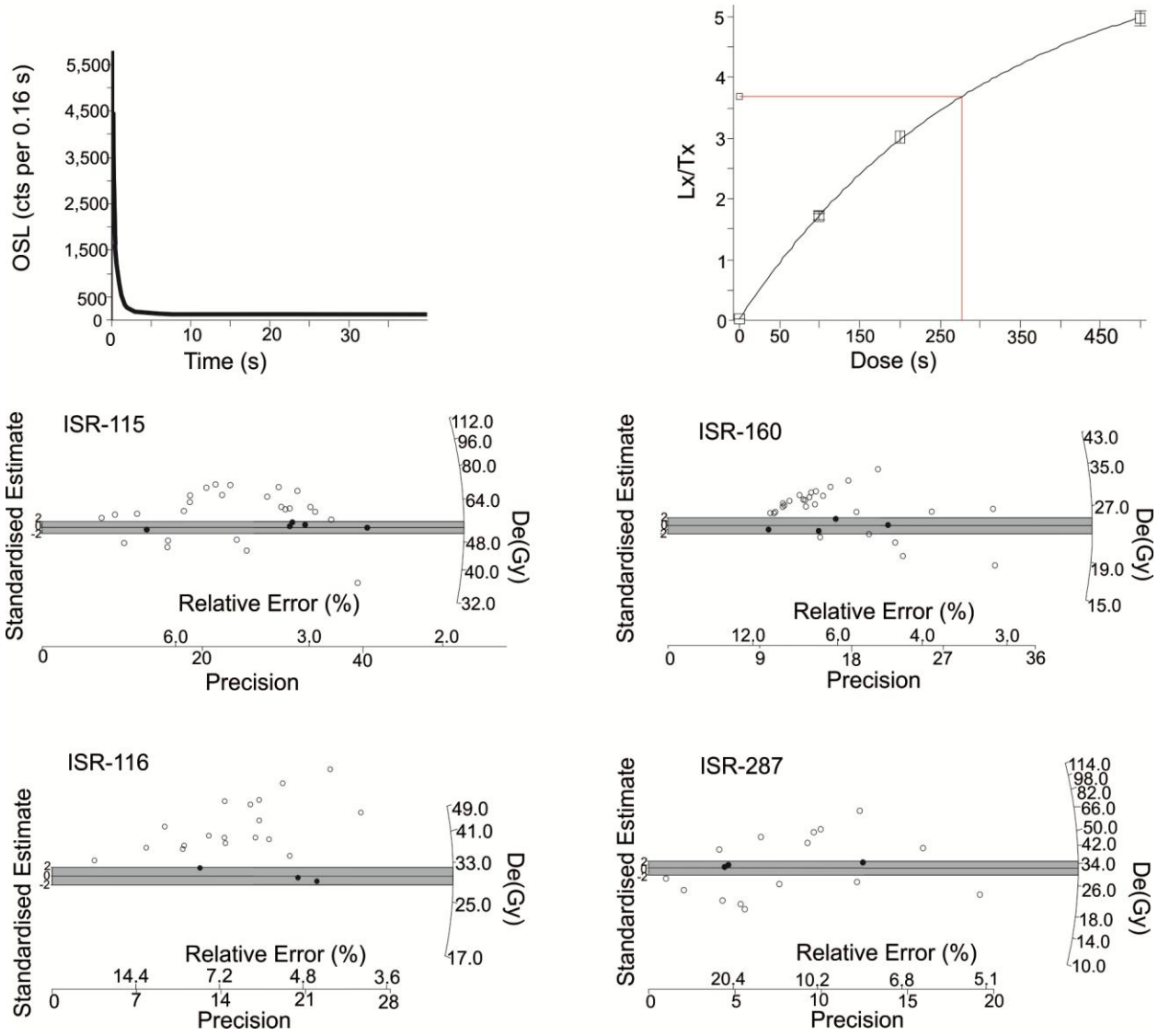

Fig. 6.3 The OSL measurements and radial plots of the De values.

Results

Table 6.1 provides the OSL ages of the Miliolite samples along with their location details. The Varli sample has yielded two distinct ages in minimum age model (MAM) and central age model (CAM). Table 6.2 list of all OSL measurements along with the concentrations of U, Th and K.

The results of OSL geochronology are discussed in comparison with the available geochronology on Kachchh as well as Saurashtra Miliolites in Chapter 7.

Sample No	Type-I, Obstacle Dune Deposit Field Reference	Latitude	Longitude	Elevation from msl	Sample collected height from surface m	Min2 sigma Age (ka)
FR-4 (ISR-115	Abandoned Quarry (Fakirwadi)	23° 11' 17"N	69° 35' 57"E	170m	0.3m	14.5± 1.3
FR-57 (ISR-116)	Abandoned miliolite quarry (Khari-catchment)	23° 10' 53"N	69° 36' 09"E	167m	0.3m	20.3± 2.1
KR-63 (ISR-160)	Roha fort Trappen Hill	23° 11' 53"N	69° 16' 26"E	200m	3.0m	8± 0.7
VR-67 (ISR-287)	Varli	23° 05' 39"N	69° 47' 39."E	145m	1.2m	4.43 ± 0.86 (MAM) 11.9 ± 1.8 (CAM)

Table 6.1 Summary of the types of Quaternary carbonate deposits and their optically stimulated luminescence age ranges in the study area.

Sample No	U (ppm)	Th (ppm)	K (%)	Cosmic ray dose rate (µGy/a)	Dose rate (µGy/a)	Weighted mean ED (gy)	Weighted mean age (ka)	Min2 sigma ED (gy)	Min2 sigma Age (ka)
FR-4	6.6 ± 0.33	8.4 ± 0.42	0.37± 0.01	196.9± 4.8	2.3± 0.2	52.5± 0.38	22.5± 2	33.71± 0.67	14.5± 1.3
FR-57	3.6 ± 0.18	4.3 ± 0.22	0.28± 0.01	196.8± 4.8	1.4± 0.1	43.45±0.63	30.8± 3	28.± 0.95	20.3± 2.1
KR-63	6.8 ± 0.34	6. 5 ± 0.33	0.36± 0.01	164.9± 37.7	2.2± 0.2	23.85± 0.23	10.7± 0.9	17.86± 0.37	8± 0.7
VR-67	3.6 ± 0.18	5.2 ± 0.26	1.19 ± .02	183.4 ± 17.5	2.2 ± 0.1	32.13 ± 4.91	11.9 ± 1.8	4.43 ± 0.8	1.6 ± 0.3

Table 6.2 U, Th, and K concentrations and optically stimulated luminescence data. Radioactivity, palaeodose, dose rate and luminescence ages (water content=10 ± 2,Juyal et al., 2006)