



## Research paper

## Methodological studies on luminescence dating of volcanic ashes

R.H. Biswas<sup>a,\*</sup>, M.A.J. Williams<sup>b</sup>, R. Raj<sup>c</sup>, N. Juyal<sup>a</sup>, A.K. Singhvi<sup>a</sup><sup>a</sup> Geosciences Division, Physical Research Laboratory, Ahmedabad 380009, Gujarat, India<sup>b</sup> Department of Geography, Environment & Population, University of Adelaide, Adelaide 5005, Australia<sup>c</sup> Department of Geology, M.S. University of Baroda, Vadodara 390 002, India

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## ABSTRACT

We report here the results of a feasibility study of luminescence dating of polymineralic volcanic ash. Of the several possible protocols that used different emission bands and different IR stimulation regimes, the post infrared-infrared stimulated luminescence (pIR-IRSL) signal (detected using a violet-blue emission window with stimulation temperature in the region 260–320 °C) provided the most stable signal. This involved, i) identification of the most suitable temperature for pIR-IRSL read out, ii) determination of alpha efficiency and, iii) estimation of anomalous (athermal) fading rate. Anomalous fading rate (g-value in %/decade) of pIR-IRSL signal at 300 °C was 0.0–1.6%/decade and it ranged from 2.4 to 5.2%/decade for IRSL at 50 °C, both preheated to 320 °C. Thus, though more stable, pIR-IRSL signals may fade in nature, and even during laboratory extended irradiation. Of the models for fading correction by Huntley and Lamothe (2001) and Kars et al. (2008), the Kars et al. (2008) model performed better as the natural luminescence intensity was closer to the onset of saturation in the luminescence dose response curve. Our measurements suggest that alpha efficiencies of the pIR-IRSL signals are higher than that of IRSL. Fading corrected pIR-IRSL single aliquot regeneration (SAR) protocol based ages on three of the five volcanic ash beds are in agreement with the expected ages of ~74 ka, based on geochemical association of the present samples to be the Youngest Toba Tuff (YTT). Other ash samples that gave ages of <24 ka and <37 ka, were inferred to have been in their secondary context, reworked from their original depositional sites. The onset of saturation dose of the pIR-IRSL signal ( $D_0$ ) was ~330 Gy and this implied a maximum measurable equivalent dose of 660 Gy. The minimum detectable dose was ~5 Gy. These dose limits correspond to a typical age range of 1–150 ka using the pIR-IRSL signal for volcanic ashes.

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## 1. Introduction

The potential of volcanic ashes as time-markers in stratigraphic sequences is well recognised, and yet the chronometry of young volcanic ashes has been difficult (Lowe, 2011). Although the use of the  $^{39}\text{Ar}/^{40}\text{Ar}$  technique provides precise and accurate ages on tephra, it is laborious and time consuming. The use of luminescence for dating volcanic materials has for long been researched and has met with only limited success due to athermal/anomalous loss of luminescence from volcanic feldspars (Wintle, 1973). Fattahi and Stokes (2003a) summarized luminescence based efforts for the chronometry of volcanic ashes that used its different mineralogical fractions such as polymineral fine grains, quartz, feldspar and glass. Thermoluminescence (TL) applications to the fine-grained (4–

11  $\mu\text{m}$ ) glass component of Mazama ash by Berger and Huntley (1983) yielded a TL age concordant with the  $^{14}\text{C}$  control ages. Berger (1985) suggested that 100% clear glass of the same samples exhibited no anomalous fading. However, glass shards physically separated from other ashes, using the same treatment did exhibit significant anomalous fading (Berger, 1985). Berger (1991, 1992), used purification of the 4–11  $\mu\text{m}$  glass shards fraction using heavy liquids and centrifuge along with an extended preheat of 50–60 °C for 8 days, and suggested that these steps ensured a TL signal that was free of athermal fading and, the TL additive-dose technique yielded accurate ages for tephra from a few hundred years to 400 ka. A routine separation of pure glass from the ash sample using these procedures was difficult and therefore Berger and Davis (1992) felt the need to remove the unstable TL signal using a pre-heat of 110 °C for 4 days. This preheating caused a dramatic reduction of equivalent dose ( $D_e$ ) corresponding to the higher temperature (~320 °C) TL and that the reduction was sample dependent. The reasons for this were not clearly understood.

\* Corresponding author. Tel.: +91 7926314369.

E-mail addresses: [biswasrabiul@gmail.com](mailto:biswasrabiul@gmail.com), [rabiul@prl.res.in](mailto:rabiul@prl.res.in) (R.H. Biswas).

Berger and Huntley (1994) analyzed the infrared stimulated luminescence (IRSL) from tephra, but the low signal to noise ratio for the luminescence signal, from a glassy matrix, restricted its use. Fattahi and Stokes (2000) reported that the rate of anomalous fading of the red TL emission from volcanic quartz was lower compared to the UV-blue emission and obtained an age of 1280 ka that accorded with the control ages. However, this work was not followed further.

In luminescence dating of volcanic ashes, a logical measurement approach would be the separation of the mineral phase with least anomalous fading (e.g. glass), isolation of the most stable luminescence signal, and dating using this signal with a proper fading correction. Separation of 100% pure glass from ash of silt and clay size grains is nontrivial and hence, is not used routinely. Further, a minor contribution from feldspar grains with their high luminescence sensitivity can mask the signal from the glass phase which has low luminescence sensitivity (Berger and Huntley, 1994).

To exploit the high luminescence sensitivity of the feldspar, we examined polymineralic ash samples and both thermoluminescence (TL) and optically stimulated luminescence (OSL) signals were examined including the post infrared-infrared stimulated luminescence (pIR-IRSL) signal (Buylaert et al., 2009, 2011; Li and Li, 2011; Thiel et al., 2011; Thomsen et al., 2008, 2011). The samples, protocols used and the results are discussed below.

## 2. Samples and locations

The samples comprised rhyolitic volcanic ash samples from river valleys across peninsular India (Jones, 2010; Jones and Pal, 2009; Williams and Royce, 1982). These deposits have been correlated geochemically to the Youngest Toba Tuff (YTT; Shane et al., 1995; Westgate et al., 1998). The  $^{40}\text{Ar}/^{39}\text{Ar}$  age of this ash is  $73 \pm 4$  ka (Chesner et al., 1991) and it is suggested that this ash event impacted ecosystems and associated hominin populations (Jones and Pal, 2009; Williams et al., 2009). At Jwalapuram (N  $15^{\circ}20'E$   $78^{\circ}10'$ ), sediments beneath and above the YTT have been optically dated to  $77 \pm 6$  and  $74 \pm 7$  ka, leading to an age of  $\sim 74$  ka for YTT (Petraglia et al., 2007). Positions of the samples in the stratigraphic sections are shown in Fig. 1 and Table 1

**Rehi section:** A 275 cm thick volcanic ash layer is located at the northern bank of Son River (N  $24^{\circ}32'30.4''$  E  $82^{\circ}16'27.1''$ ). Two ash

**Table 1**  
Stratigraphy, sample details, and the position of the samples.

Sites	Thickness of ash layer (cm)	Sample	Type	Sample position
Rehi	275	RS05 1-4	Clay rich sand	15 cm above the top of the ash
		SON05	Ash	200 cm above the ash base
		Rehi 2-1		
		RS05 1-3	Ash	25 cm above the ash base
		RS05 1-2	Coarse sand	10 cm beneath the ash base
		SON05	Coarse sand	25 cm beneath the ash base
Kuntheli	183	Rehi 2-2		
		K05 1-3	Ash	120 cm above the ash base
		SON05 K 1-3	Ash	65–80 cm above the ash base
		SON05 K 1-2	Sandy loam	65 cm beneath the ash base
Tejpur	10–20	TPS-1	Sandy silt	10 cm above the ash
		TPASH	Ash	Center of 10–20 cm layer
		TPS-2	Silty sand	30 cm beneath the ash base
Bori	~50	Bori	Ash	Center of ash layer
Morgaon	~50	Morgaon	Ash	Center of ash layer

samples and three samples of sediments, overlying and underlying the ash bed were collected (Williams et al., 2009).

**Kuntheli section:** This site is located at the southern bank of Son River (N  $24^{\circ}30'10.5''$  E  $82^{\circ}00'59.4''$ ). Thickness of the ash layer at this site was 183 cm. Two ash samples and a sediment sample underlying the ash layer were collected (Williams et al., 2009).

**Tejpur section:** An ash layer at the right bank of the River Madhumati near Tejpur (N  $21^{\circ}45'12''$  E  $73^{\circ}18'29''$ ) associated with valley fill terraces was sampled. Thickness of the ash layer at this site was  $\sim 10$ – $20$  cm. An ash sample and a sample each from overlying and underlying sediment were collected for analysis (Raj, 2008).

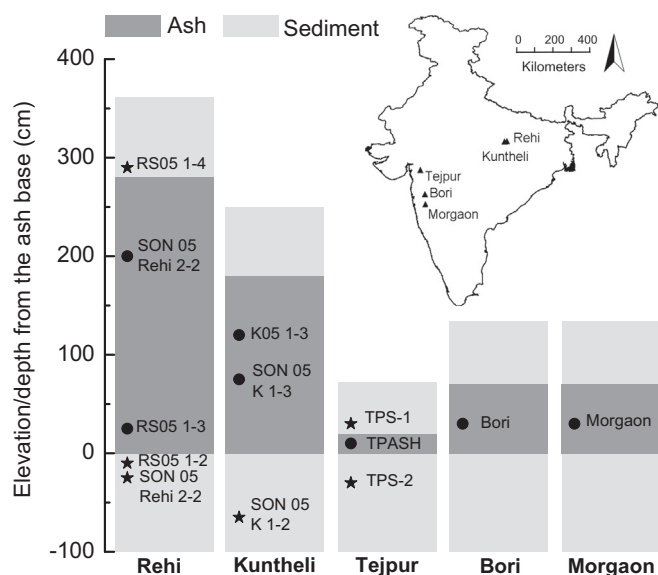
**Bori section:** The ash here is situated along the Kukdi River near the village Bori (N  $19^{\circ}17'30''$  E  $74^{\circ}06'30''$ ). The thickness of the deposit varies from 0.2 m to 1 m and a sample from the middle of the ash bed was collected (Korissettar et al., 1989).

**Morgaon section:** The site is located on the left bank of the river Karha (N  $18^{\circ}16'40''$  E  $74^{\circ}09'29''$ ). The thickness of the ash layer is  $\sim 50$  cm and a sample from the middle of the ash bed was collected (Mishra et al., 2009).

The maximum preserved thickness of the YTT in marine cores in the Bay of Bengal is  $\sim 10$ – $15$  cm (Gasparotto et al., 2000; Ninkovich, 1979; Ninkovich et al., 1978a, 1978b). So it is likely that the original air fall ash mantle deposited across peninsular India was also of a similar thickness. Erosion of the ash would have begun soon after deposition, with ash moving down slope into depressions and river channels under the combined influence of slope wash, soil creep and rill erosion. We consider that the YTT outcrops in India were almost certainly reworked to some degree, possibly more than once and therefore although we accessed the same sample, these had variable post depositional histories providing a good range to test the validity of the luminescence dating of volcanic ashes.

## 3. Experimental details

The samples were collected from freshly exposed sections and in specially designed aluminium pipes (Chandel et al., 2006). For ash samples, equivalent doses were measured using the polymineralic fine grain (4–11  $\mu\text{m}$ ) technique (Zimmerman, 1971). Samples were treated with 1N HCl, and 30%  $\text{H}_2\text{O}_2$  and then deflocculated using



**Fig. 1.** Location, stratigraphy and position of the samples.

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