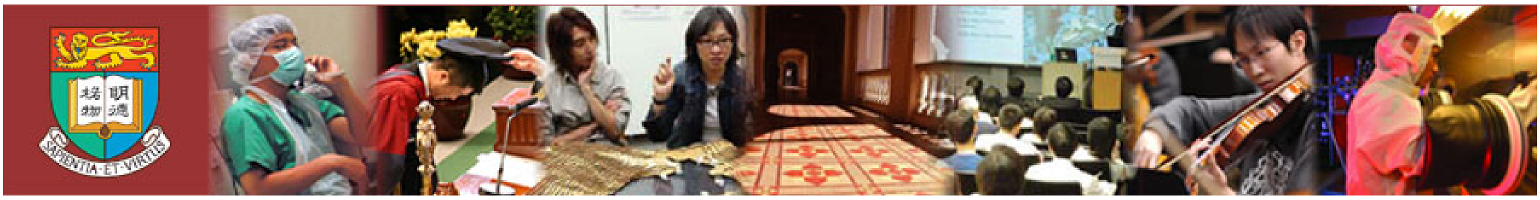

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Luminescence Dating of Volcanic Eruptions in Datong, Northern China

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Abstract: In this study, fine-grain quartz was used for luminescence dating for lava baked samples from different sites in Datong. Optical stimulated luminescence (OSL), thermal transferred OSL (TT-OSL)/recuperated OSL (Re-OSL) and thermoluminescence (TL) dating protocols were applied. For these samples, the OSL signals saturate at about 300-400 Gy, which limits their age to less than 100 ka based on their ambient dose rates. The TT-OSL/Re-OSL method has poor dose recovery. TL dating gives reliable results, and multiple-aliquot regenerative-dose TL method with sensitivity change correction based on the 325 ºC TL of a test dose can be applied for samples up to 400 ka. The results indicate that the ages of the volcanoes in Datong are from 380 ka to 84 ka. The volcanic activity started earlier in the southeast area than those in the northwest part, which is consist with the literature data.

Key words: Datong volcanoes; Thermoluminescence dating; Fine grain quartz

1. Introduction

The volcanic groups in Datong, northern China, with volcanic eruptions lying between Quaternary loess layers or lacustrine sediments, provide unique material for both volcanic and Quaternary studies. Previous studies have been carried out in this area (Pei, 1982; Liu, et al., 1983; Hurford and Chen, 1986; Zhu, et al., 2006; Lei, et al., 2013). The ages of eruptions can provide crucial information for geological studies, such as giving age information for tectonic activity. Besides, dating the volcanic eruptions contributes to the understanding of cycles and the temporal trends of volcanic activities, which helps to forecast volcanic disasters.

Different dating methods such as K-Ar dating, thermoluminescence (TL) dating on poly-minerals and palaeomagnetic dating were applied to materials from various Datong volcanic sites. Substantially different ages were given by different dating methods (Li and Sun, 1984; Li, 1988; Zhu et al., 1990; Chen et al., 1992). For most samples, the ages given by using TL method were younger than using K-Ar dating method with obvious differences, e.g., on site of Yujiazhai, 37 ka or 41 ka for K- Ar dating method (Li, 1988; Chen et al., 1992) and 29 ka for TL dating method (Li and Sun, 1984). Due to the difference on the ages, different start time, ending time and phases for the eruptions of the Datong volcanoes were reported (Yin, 1976; Li et al, 1982; Chen et al., 1992). Based on the stratigraphy of volcanic sequences, Yin (1976) concluded that the volcanic activities started in the middle-late Middle Pleistocene (about 300-400 ka) and terminated in the early Late Pleistocene (about 150 ka) in the southeast area, while in the northwest area, the volcanoes erupted since the early Late Pleistocene (about 150 ka), ending in the late Late Pleistocene (about 100 ka). However, Chen et al. (1992) using the K-Ar dating method concluded that the onset of the volcanic eruptions in the Datong volcanic area was about 0.74 Ma in the south area. In the period of about 400 ka ago, volcanic activities spread all over the volcanic area, and later the eruption center turned to south area again at about 0.20 Ma ago. The ages of volcanic eruptions in Datong and the division of volcanic phases are still in controversial. Luminescence dating methods have developed rapidly in recent 20-30 years, after the previously reported ages. Single aliquot regenerative-dose (SAR) method, thermally transferred optically stimulated luminescence (TT-OSL) method and recuperated OSL (Re-OSL) method were proposed (Murray and Wintle, 2000; Murray and Wintle, 2003; Duller, 2004; Wang et al., 2006; Adamiec et al., 2010; Berger, 2011; Ganzawa and Ike, 2011; Duller and Wintle, 2012; Li, et al., 2013). The application of using luminescence methods to volcanic material and materials baked by volcanic activity have also been researched by various other authors (Miallier et al.,1994; Li and Yin, 2001; Fattahi and Stokes, 2003; Ganzawa et al., 2005;

Bassinet, 2006; Tsukamoto et al., 2007; Shitaoka et al., 2014).

Zhao et al. (2012) has applied the OSL method to the samples from Datong volcanic area. They concluded that the volcanoes in Dongshuitou erupted at about 8.5 ka, and volcanoes in Yujiazhai erupted more than 170 ka ago. Ages obtained by using this method may be underestimated because of signal saturation for the old samples, so some ages they obtained were considered as the minimum ages. TT-OSL and Re-OSL methods were demonstrated to be saturated at larger doses and more suitable for dating of older samples than OSL signals (Wang et al., 2006; Adamiec et al., 2010). Besides, TL signals are more stable than OSL signals and may be more proper for dating older deposits. Kusiak et al. (2013) have confirmed the possibility of TL dating method for obtaining ages older than 400 ka.

In this paper, we report the dating results using OSL, TT-OSL/Re-OSL and TL dating protocols for lava baked samples from different sites in Datong.

2. Sample collection and preparation

Datong volcano group is located in the east of Datong Basin. Volcanoes in this area erupted in several phases, leading to the existence of several eruption layers in one place. Based on the type of volcanic eruption and the difference of underlying layers, the Datong volcanic area was divided into two parts. In the northwest of Chenzhuang-Xubao fault, it is called the north area. The volcanoes in this area were explosive erupted, and volcanic cones were formed. The heights of the volcanic bodies are ranging from 20 m to 150 m. The underlying layer in this area is loess. In the southeast, the area is named south area. The eruptions of volcanoes were relatively quiet, and lava flows are overlying the lacustrine layers. The thickness of the lava in the south area is ranging from about 1m to more than 10 m. The volcanic eruptions in both parts are basaltic. The loess and the lacustrine sediments below the lava flows layer in a distance within about 60-80 cm were red in different degrees, because of being heated by lava above (Li and Sun, 1984).

In this study, samples were collected from six different sites from Datong volcano groups (Fig. 1).Samples in this paper were collected in the loess or lacustrine layers within a distance of 30 cm below the volcanic eruptions. Blocks of the baked samples were collected and kept in the black plastic bags.

All the samples collected were prepared in the laboratory under dim red light conditions. After chipping away the outer 10 mm of the sampled blocks by knife, the remaining samples were treated with 30% H_2O_2 and 30% HCL to remove the organic matter and the carbonates, respectively. Then, they were treated with 30% fluorosilicic acid (H_2SiF_6) for 5 days for the removal of the feldspar contamination. Infrared (IR) stimulation was performed to check the purity of the quartz (Li et al., 2002). If the IRSL signal intensity was 3 times larger than the background, which indicates the presence of feldspar, a second treatment with H_2SiF_6 would be carried out. The extracted fine grains in the range of 4-11 μm were selected using physical separation method based on Stokes' Law and then deposited from suspension in water on stainless steel discs.

3. Equipment and measurements

3.1 Equipment

The luminescence measurements in this experiment were performed using a Daybreak 2200 TL/OSL automatic system. A combined blue (470±5 nm) and infrared (880±80 nm) LED OSL units and A $^{90}Sr/^{90}Y$ beta source, delivering ~0.05 Gy/s, were equipped for irradiation. All the luminescence signals were detected by an EMI 9235QA photomultiplier tube through two 3mm U-340 filters. The OSL measurements were made at 125 ºC with blue stimulation power set at ~45 mW cm^{-2}. In TL measurements, the heating rate was 5 ºC/s. A SX2-10-13 oven with the max temperature of 1350 ºC was used for annealing the samples and a solar lamp (SOL2) was used for bleaching the samples.

3.2 OSL dating method

OSL dating of multiple-aliquot regenerative dose method as proposed by Wang (2005) was applied to the samples. Sensitivity changes were corrected by the signals of the test dose after each OSL measurement. The integration of the first 5 seconds minus that of the last 5 seconds of 100 s measurement was used for equivalent dose (De) calculation. Aliquots were annealed at 500 ºC for 10 min in the oven before administrating the regenerative doses.

Dose recovery test was carried out using sample YJZH-1. The sample with a known given dose (GD) of 261 Gy can be recovered well with the recovery dose (RD) of 243±10 Gy and the dose recovery ratio (DRR) of 0.93 (Fig. S1a). However, the sample given the known dose of 1046 Gy was poorly recovered with the ratio of 1.17 (Fig. S1b). This may be due to the saturation of the growth curve, which leads to the underestimation of the equivalent doses. Therefore, we concluded that the OSL method might be limited to samples with De of less than about 300-400 Gy, which corresponds for these samples to ages of <100 ka, in consideration of ambient dose rate.

3.3 TT-OSL/Re-OSL dating method

Natural TT-OSL and the Re-OSL signals were examined for samples YJZH-1 and DSTOSL-2. The TT-OSL and Re-OSL signals can be observed, although they were about 50 times weaker than OSL signals. Aliquots of sample YJZH-1 were bleached for 1 h by the solar lamp. Different regenerative doses were given. The TT-OSL/Re-OSL increases with regenerative doses and growth curves can be constructed (Fig. S2).

Dose recovery test was carried out using sample YJZH-1. Regeneration aliquots were obtained by bleaching under solar lamp for 1 h and administrating different regenerative doses. We selected two different protocols for the dose recovery test. One was the Re-OSL procedure Wang et al. (2006) applied for loess and the other was the TT-OSL protocol proposed by Liu et al. (2012). The detailed processes of the two approaches are displayed in

Table S1.

The samples were given a dose of 0 Gy, 531 Gy, 632 Gy, 1264 Gy respectively after bleaching. The dose recovery results using the Re-OSL protocol A (Wang et al., 2006) are shown in Fig. S2a, b, and those using the TT-OSL protocol B（Liu et al., 2012) are shown in Fig. S2c, d. Both of the protocols yield poor dose recovery with the recovery error more than 40%, indicating that the TT-OSL and Re-OSL method cannot accurately recover the sample. The other samples from the Datong volcanic areas have the similar property on luminescence signals, therefore we consider the TT-OSL and Re-OSL method maybe not proper on other samples either.

3.4 TL dating method

In the TL dating, the signals in the ‘stable’ region of the glow curve should be used for calculation. This region can be identified by means of the plateau test (Aitken, 1985). In this experiment, the curve of the ratio between natural and artificial dosed glow-curves versus temperature, called the plateau curve, was derived to get the temperature range for dating. The plateau curve for sample YJZH-1 is demonstrated in Fig. S3. From the figures, we considered that the TL data ranging from 300 ºC to 360 ºC was used for dating.

The reliability of the TL dating method was examined through the dose recovery test using the sample YJZH-1. Two approaches were applied for zeroing for the regeneration aliquots. One way was to bleach by sunshine, and the other was to anneal in the oven.

We have studied the zeroing of the signals by bleaching and annealing. For bleaching, the samples were bleached by sunlight in for 28 hours in Hebei, China, and then given the recovery doses or the regenerative doses. In the dose recovery test, the TL measurements of all the regeneration aliquots and recovered aliquots were carried out at 500 ºC for 60s after preheating at 260 ºC for 10 s. Growth curve and the equivalent dose were obtained (Fig. 2). The sample with given dose of 612.7 Gy was recovered as 501±12 Gy, with the recovery ratio of 0.82, indicating that this method is unable to recover the doses. In addition, we observed that the TL signals were not bleached to zero completely. The residual signal may have influences on the dose recovery results.

For zeroing the signals by annealing, the samples were annealed at 500 ºC for 10 minutes in the oven, and then cooled down naturally. Thereafter, the known recovery doses or the regenerative doses were administered to the samples. Many studies have shown that the luminescence sensitivity changes after anneal (Han et al., 2000; Chen et al., 2001). We tried to correct it by using both the 110 ºC TL response to a test dose before the TL measurement (step 4 in Table S2, corrected as $L/T_{110\ ^{\circ}C}$) and the 325 ºC TL peak response to a subsequent dose after TL measurements (step 8 in Table S2, corrected as L/T). The detailed procedures for each disc are shown in Table S2. The results of the dose recovery tests are shown in Fig. 3. Using the method of correcting by 110 ºC TL peak, doses could be recovered with the recovery ratio of about 0.89, but the spots went away from the fitted curves (Fig. 3a, b), which may be attributed to the unstable of 110 ºC TL signals. However, using the method of correcting by 325 ºC TL peak, the dose recovery test yielded acceptable values. The dose recovery ratio was 0.97 for the samples of about 200 ka and 0.88 for the samples of about 400 ka, within the allowable limits. Besides, there is no sign of saturation in the regenerative dose range. Therefore, we adopted 325 ºC TL corrections in the estimation of the equivalent dose in TL dating measurements for all the samples from the baked layers in Datong.

4. Dating results and discussions

4.1 Estimation of equivalent dose

Different luminescence methods were applied to determining the equivalent dose of the volcanic samples from Datong. From the dose recovery test, OSL can only recover the dose for samples younger than 100 ka, due to the ambient dose rate and the signal saturating at about 300-400 Gy. For the samples older than 100 ka, the growth curve reaches saturation. Using the TT-OSL and Re-OSL protocols, the doses cannot be recovered. This may be related to the large pre-dose of the samples (Li and Li，2006) .

The multiple-aliquot regenerative-dose TL method was carried out. The TL sensitivity does not change significantly after bleaching. However, the known dose cannot be recovered, which may be due to the TL residual signal. Considering that the samples from the Datong volcanoes had experienced heating, the TL was zeroed in the past, annealing at 500 ºC was applied on the samples for zeroing. After annealing, the TL sensitivity changed distinctly, so sensitivity corrections were conducted. The sensitivity correction by the 325 ºC TL response to a subsequent test dose gave reliable results. The dose response curves can be well fitted with a saturating exponential function. No sign of signal saturation was observed in the regenerative dose range.

Based on the dose recovery result, the equivalent doses of all the samples from baked layers in Datong were determined by the regenerative-dose TL method. The equivalent doses obtained are shown in Table 1 and Fig. 4.

Dongshuitou and Yujiazhai sites of the volcanoes have been dated using the additive dose multiple aliquot TL method of polyminerals about 30 years ago (Li and Sun, 1984). The equivalent doses obtained by these authors are about 200-300 Gy larger than the results from this study. This has given rise to the different measurement procedures used. The additive-dose TL method might lead to overestimation as the linear extrapolations in the growth curves were applied in their studies. No extrapolation was applied in the regenerative-dose TL method used in this study, but sensitivity correction is crucial when using this method. Inadequate sensitivity correction may lead to unacceptable ages.

4.2 Estimation of dose rate

The dose rates were calculated from the U, Th and K concentrations of samples. We have also taken into account for the effects of water content and contribution from cosmic rays (Aitken, 1985). The U, Th and K were measured by ICP-MS (Inductively coupled plasma mass spectrometry). The water content was estimated based on the wet and dry weight of samples measured in the laboratory. The contribution of cosmic ray was mainly assessed based on the depth of samples. Dose rate values of all the samples are showed in Table 1.

4.3 The eruption ages of volcanoes in Datong, northern China

In the literature, both relative ages and absolute ages of volcanic eruptions in this area were reported. The main methods used for determining the ages were paleomagnetic dating method (Zhu et al., 1990), relative age successions based on the stratigraphy of volcanic sequences (Yin, 1976), K-Ar dating method (Zhu et al., 1990; Chen et al., 1992), and luminescence dating method (Li and Sun, 1984; Li, 1988; Zhao et al., 2012). In these results, K-Ar dating method gave older ages than others did. It may be due to the atmospheric excess argon (Chen et al., 1992), so we would not consider the K-Ar ages in the comparison below.

TL ages we have obtained (summarized in Table 1) are compared with ages determined by other methods in Datong volcanic sites. In the north area, volcanic eruptions in Dongshuitou were dated at about 84 ka ago, which is similar to the results from OSL method (Zhao et al., 2012). It is younger than 120 ka reported by Li and Sun (1984) using additive dose TL method. In the Xigelao Mountain site, we measured the volcanic eruption to have an age of about 256 ka. In the south area, different eruption ages in Yujiazhai site were reported. The TL result was about 290 ka (Li and Sun, 1984) and OSL age more than 170 ka (Zhao et al., 2012). Using the TL method we proposed, the age is 270 ka, a little younger than the TL age by Li and Sun (1984), and does not contradict the OSL result. For other sites in the south area, data of absolute ages for volcanic eruptions are lacking. By using the TL method in this paper, it indicated that the volcanoes in Nanshi Mountain area erupted dating back to about 380 ka, the volcanoes in Yujiaxiaobao erupted dating back to about 220 ka, and the volcanoes in Xishawo erupted dating back to about 192 ka. From the comparisons, we noticed that the ages obtained by Li and Sun (1984) using TL method were larger than our results. This might be attributed to the difference in procedures for getting the equivalent dose, as discussed in section 4.1.

The TL method we proposed recovered well in the dose recovery test and the ages derived from the procedure we proposed were reliable. Based on the TL results we obtained using the improved TL method, there were more than 4 phases of volcanic eruptions in the volcanic area. Volcanoes in Nanshi Mountain erupted dating back to about 380 ka. Volcanoes in Xigelao Mountain and Yujiazhai erupted dating back to about 260 ka. Volcanoes in Yujiaxiaobao and Xishawo erupted dating back to about 200 ka. Volcanoes in Dongshuitou erupted dating back to about 85 ka.

Besides the ages of the volcanoes we studied, some other volcanic eruption ages in the northern part of the Datong volcanic area were also reported. The eruption age in Huangjiawa was reported as 98 ka using TL method (Li and Sun, 1984). For Hei Mountain, Li (1988) obtained TL ages of 210 ka, 170 ka, 150 ka for three lava layers; Li and Sun (1984) got the TL ages of 170 ka for one lava layer; and Zhu et al. (1990) considered the eruption ages of two lava layers as 160 ka, 100 ka, using paleomagnetic dating method.

The results showed that as a whole, the volcanic eruptions in the southeast area started earlier than those in the northwest part, which is consist with the conclusions of Yin (1976). The oldest eruption age is about 380 ka from this study. In the south area, the volcanic activity was from about 380 ka to about 200 ka; and in the north area, it started at about 260 ka and terminated at about 85 ka. The volcanic eruptions were frequent during about 260 ka to about 150 ka. We would point out that the volcanoes in different places erupted in different time and there may be several eruptions just in one place. In addition, it is possible that there were samples in other places older or younger than the ones from places where we collected for the measurement. Therefore, further ages should be measured in more places to confirm the onset of the volcanic eruptions in Datong volcanic area.

5. Conclusions

Compared with the OSL, TT-OSL and Re-OSL method, the TL method may be more suitable for dating such samples from the Datong volcanic field, especially for samples with ED of more than about 300-400 Gy, which corresponds to ages of older than 100 ka. The TL method we applied was a regenerative-dose method instead of the additive-dose TL method Li and Sun (1984) used. Annealing for zeroing and sensitivity correction by the 325 ºC TL response to the subsequent test dose was carried out, and dose recovery tests were successful. Using the TL method we proposed, at least 4 phases of volcanic eruptions were revealed in the Datong volcanic area.

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Table 1

TL dating results of samples from volcanic baked layers from different sites in Datong

Lab No.	Distance from lava (cm)	U (ppm)	Th (ppm)	K (ppm)	Water content (%)	Dose rate (Gy/ka)	De (Gy)	OSL age (ka)	Sample site
YJXB-OSL-1	15	1.29	7.61	1.31	3.80	2.64±0.1	573.8±42.7	217.4±18.4	Yujiaxiaobao
XSW-OSL-1	30	1.21	7.16	1.69	0.47	3.03±0.1	580.7±43.1	191.7±16.2	Xishawo
NSS-OSL-1	15	0.99	7.04	1.91	0.19	3.16±0.1	1205.8±198.0	381.6±64.5	Nanshi Mountain
XGLS-OSL-1	5	1.58	9.74	1.93	1.31	3.60±0.1	921.2±15.0	255.9±11.1	Xigelao Mountain
YJZH-1	10	1.57	7.57	1.86	0.94	3.26±0.1	874.0±21.9	268.1±12.7	Yujiazhai
DSTOSL-2	15	2.18	10.7	2.11	6.25	4.01±0.2	337.4±18.0	84.1±5.6	Dongshuitou

Figure captions:

Fig. 1. Sites of samples collected from Datong , China. (Δ) is the sampling sites.

Fig. 2. Results of dose recovery test using TL method for sample YJZH-1. The regeneration aliquots were zeroed by bleaching.

Fig. 3. Results of dose recovery test using (a, b) TL method corrected by 110 ºC TL peak and (c, d) TL method corrected by 325 ºC TL peak. The given doses are about 613 Gy for (a, c) and about 1225 Gy for (b, d). The regeneration aliquots were zeroed by annealing.

Fig. 4. TL growth curves for samples from volcanic baked layers from different sites in Datong.

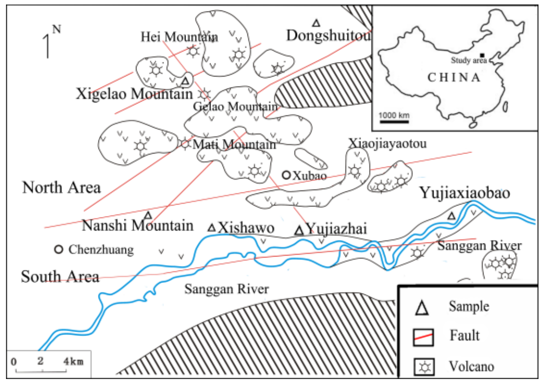
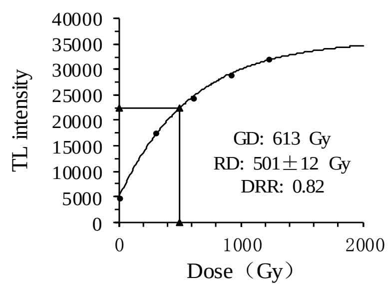

N
Hei Mountain
Dongshuitou
CHINA
Study area
1000 km
Xigelao Mountain
Gelao Mountain
Mati Mountain
Xiaojiayaotou
Xubao
North Area
Yujiaxiaobao
Nanshi Mountain
Xishawo
Yujiazhai
Chenzhuang
Sanggan River
South Area
Sanggan River
Sample
Fault
Volcano
0 2 4km

Fig. 1.



TL intensity
40000
35000
30000
25000
20000
15000
10000
5000
0
0
1000
2000
Dose（Gy）
GD: 613 Gy
RD: 501±12 Gy
DRR: 0.82

Fig. 2.

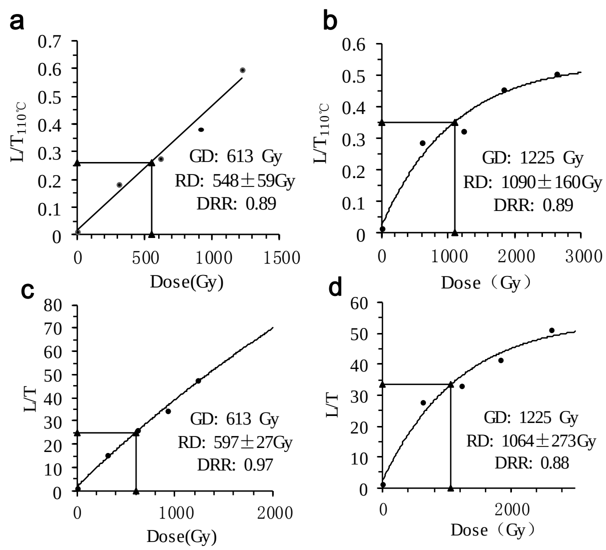

a
L/T110℃
Dose(Gy)
GD: 613 Gy
RD: 548±59Gy
DRR: 0.89
b
L/T110℃
Dose（Gy）
GD: 1225 Gy
RD: 1090±160Gy
DRR: 0.89
c
L/T
Dose(Gy)
GD: 613 Gy
RD: 597±27Gy
DRR: 0.97
d
L/T
Dose（Gy）
GD: 1225 Gy
RD: 1064±273Gy
DRR: 0.88

Fig. 3.

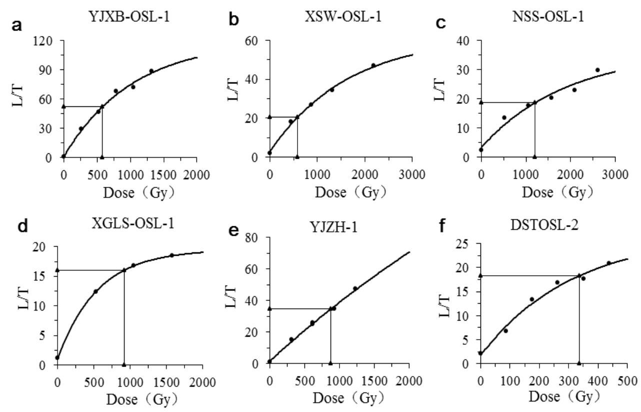
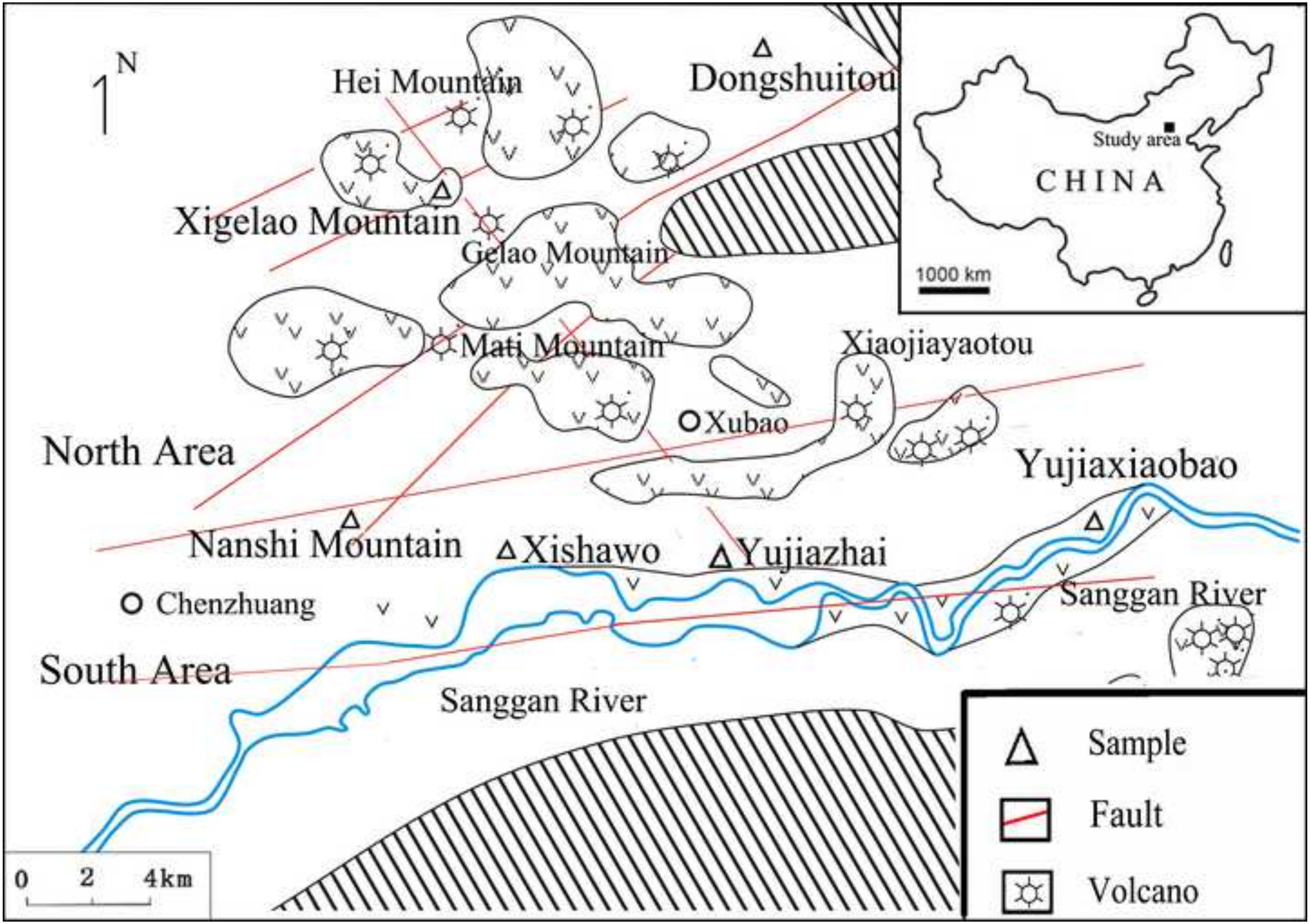

a
YJXB-OSL-1
b
XSW-OSL-1
c
NSS-OSL-1
d
XGLS-OSL-1
e
YJZH-1
f
DSTOSL-2
L/T
Dose（Gy）

Fig. 4.


N
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Supplementary Data

Click here to download Supplementary Data: Supplementary data.doc