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# Luminescence Dating of Volcanic Eruptions in Datong, Northern China

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## 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.

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

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

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

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

## 54 2. Sample collection and preparation

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

64 being heated by lava above (Li and Sun, 1984).

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

68 All the samples collected were prepared in the laboratory under dim red light conditions. After chipping away  
69 the outer 10 mm of the sampled blocks by knife, the remaining samples were treated with 30% H<sub>2</sub>O<sub>2</sub> and 30% HCL  
70 to remove the organic matter and the carbonates, respectively. Then, they were treated with 30% fluorosilicic acid  
71 (H<sub>2</sub>SiF<sub>6</sub>) for 5 days for the removal of the feldspar contamination. Infrared (IR) stimulation was performed to check  
72 the purity of the quartz (Li et al., 2002). If the IRSL signal intensity was 3 times larger than the background, which  
73 indicates the presence of feldspar, a second treatment with H<sub>2</sub>SiF<sub>6</sub> would be carried out. The extracted fine grains in  
74 the range of 4-11 μm were selected using physical separation method based on Stokes' Law and then deposited  
75 from suspension in water on stainless steel discs.

## 76 3. Equipment and measurements

### 77 3.1 Equipment

78 The luminescence measurements in this experiment were performed using a Daybreak 2200 TL/OSL  
79 automatic system. A combined blue (470 ± 5 nm) and infrared (880 ± 80 nm) LED OSL units and A <sup>90</sup>Sr/<sup>90</sup>Y beta  
80 source, delivering ~0.05 Gy/s, were equipped for irradiation. All the luminescence signals were detected by an EMI  
81 9235QA photomultiplier tube through two 3mm U-340 filters. The OSL measurements were made at 125 °C with  
82 blue stimulation power set at ~45 mW cm<sup>-2</sup>. In TL measurements, the heating rate was 5 °C/s. A SX2-10-13 oven  
83 with the max temperature of 1350 °C was used for annealing the samples and a solar lamp (SOL2) was used for  
84 bleaching the samples.

## 85 3.2 OSL dating method

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

91 Dose recovery test was carried out using sample YJZH-1. The sample with a known given dose (GD) of 261  
92 Gy can be recovered well with the recovery dose (RD) of  $243 \pm 10$  Gy and the dose recovery ratio (DRR) of 0.93  
93 (Fig. S1a). However, the sample given the known dose of 1046 Gy was poorly recovered with the ratio of 1.17 (Fig.  
94 S1b). This may be due to the saturation of the growth curve, which leads to the underestimation of the equivalent  
95 doses. Therefore, we concluded that the OSL method might be limited to samples with  $D_e$  of less than about  
96 300-400 Gy, which corresponds for these samples to ages of <100 ka, in consideration of ambient dose rate.

## 97 3.3 TT-OSL/Re-OSL dating method

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

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

106 Table S1.

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

### 113 3.4 TL dating method

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

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

122 We have studied the zeroing of the signals by bleaching and annealing. For bleaching, the samples were  
123 bleached by sunlight in for 28 hours in Hebei, China, and then given the recovery doses or the regenerative doses.  
124 In the dose recovery test, the TL measurements of all the regeneration aliquots and recovered aliquots were carried  
125 out at 500 °C for 60s after preheating at 260 °C for 10 s. Growth curve and the equivalent dose were obtained (Fig.  
126 2). The sample with given dose of 612.7 Gy was recovered as  $501 \pm 12$  Gy, with the recovery ratio of 0.82,

127 indicating that this method is unable to recover the doses. In addition, we observed that the TL signals were not  
128 bleached to zero completely. The residual signal may have influences on the dose recovery results.

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

## 142 4. Dating results and discussions

### 143 4.1 Estimation of equivalent dose

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



148 related to the large pre-dose of the samples (Li and Li, 2006) .

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

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

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

#### 165 4.2 Estimation of dose rate

166 The dose rates were calculated from the U, Th and K concentrations of samples. We have also taken into  
167 account for the effects of water content and contribution from cosmic rays (Aitken, 1985). The U, Th and K were  
168 measured by ICP-MS (Inductively coupled plasma mass spectrometry). **The water content was estimated based on**

169 the wet and dry weight of samples measured in the laboratory. The contribution of cosmic ray was mainly assessed  
170 based on the depth of samples. Dose rate values of all the samples are showed in Table 1.

#### 171 4.3 The eruption ages of volcanoes in Datong, northern China

172 In the literature, both relative ages and absolute ages of volcanic eruptions in this area were reported. The  
173 main methods used for determining the ages were paleomagnetic dating method (Zhu et al., 1990), relative age  
174 successions based on the stratigraphy of volcanic sequences (Yin, 1976), K-Ar dating method (Zhu et al., 1990;  
175 Chen et al., 1992), and luminescence dating method (Li and Sun, 1984; Li, 1988; Zhao et al., 2012). In these results,  
176 K-Ar dating method gave older ages than others did. It may be due to the atmospheric excess argon (Chen et al.,  
177 1992), so we would not consider the K-Ar ages in the comparison below.

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

190 as discussed in section 4.1.

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

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

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

## 210 5. Conclusions

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

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295 Table 1

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

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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

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312 **Figure captions:**

313 Fig. 1. Sites of samples collected from Datong , China. ( $\Delta$ ) is the sampling sites.

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

315 Fig. 3. Results of dose recovery test using (a, b) TL method corrected by 110 °C TL peak and (c, d) TL method corrected by 325 °C TL  
316 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.

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

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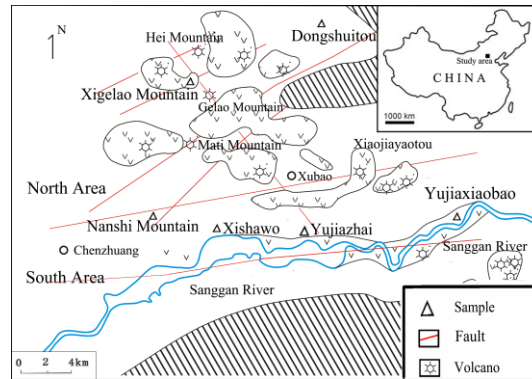
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338 Fig. 1.

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357 Fig. 2.

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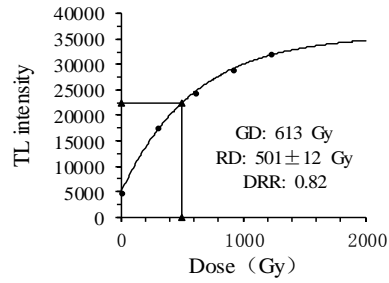
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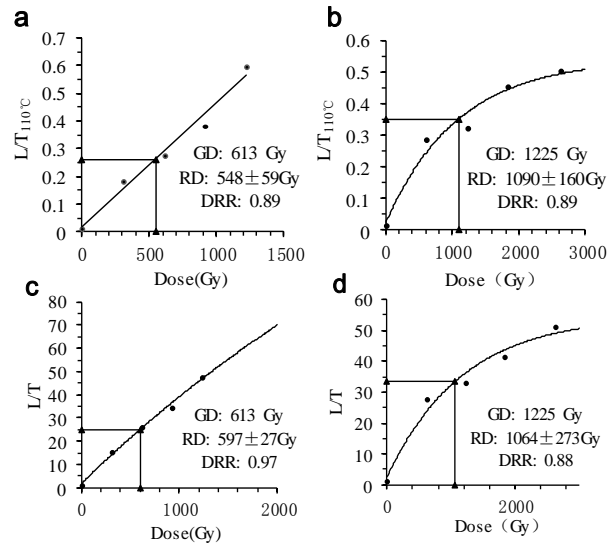


Fig. 3.

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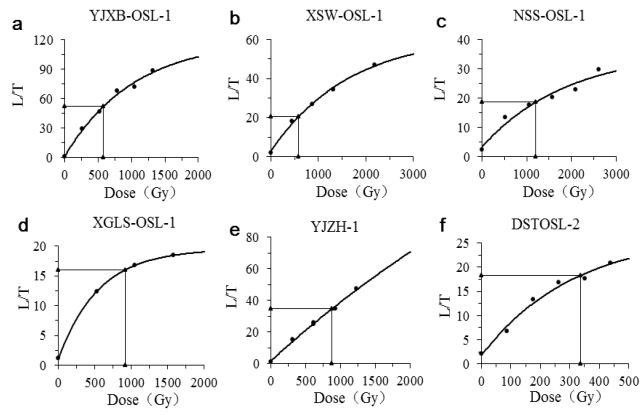
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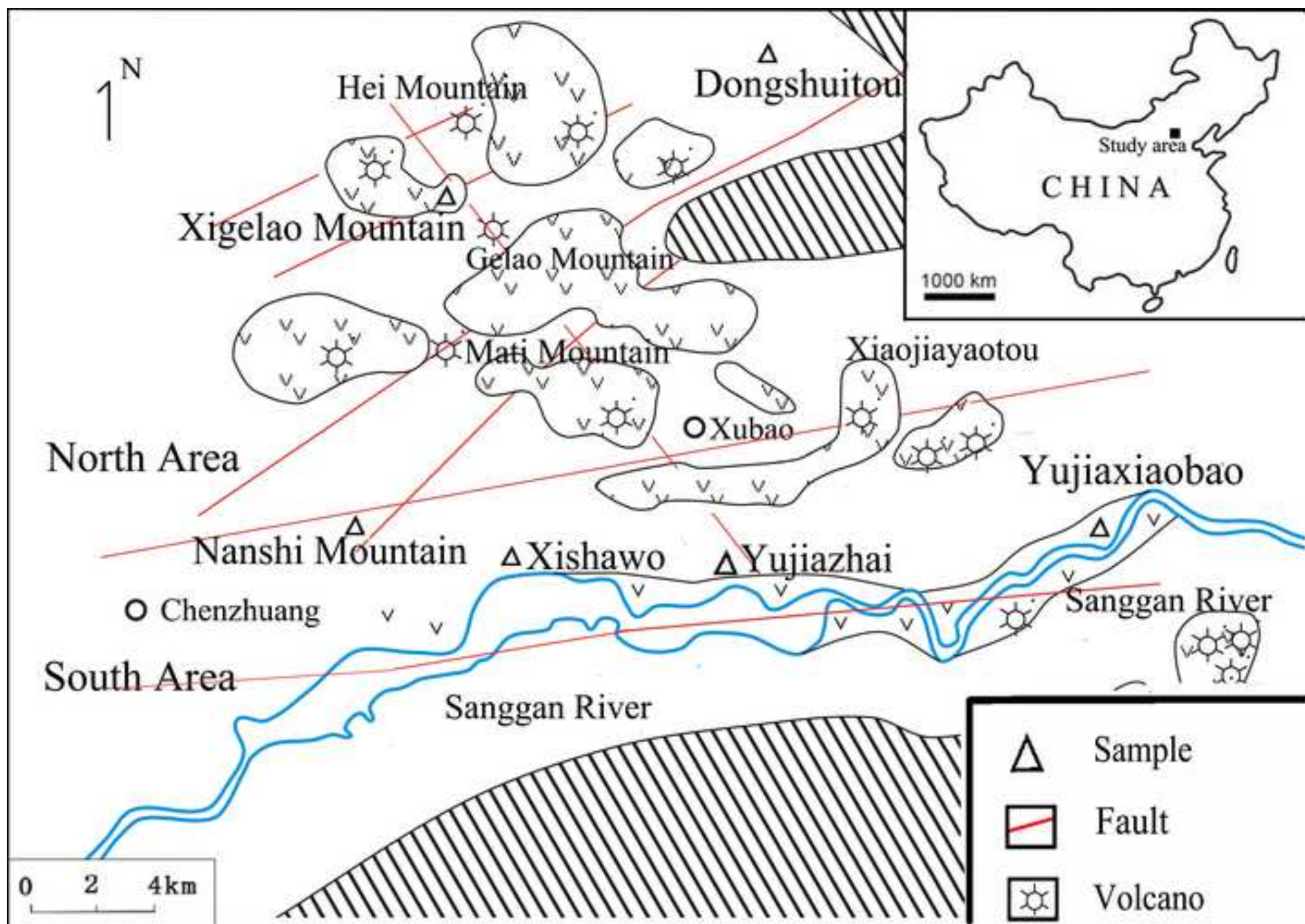
401 Fig. 4.

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Figure

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**Supplementary Data**

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