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Clay Li and Nd isotopes response to hydroclimate changes in the Changjiang (Yangtze) basin over the past 14,000 years

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1 **Abstract**

2 In large river basins, the relationship between silicate weathering and climate
3 variability on various temporal scales is still highly debated. This study presents clay
4 Li-Nd isotopes and elemental compositions of bank sediments from the Changjiang
5 (Yangtze) River, and of sediments from core CM97 recovered from its delta. In the
6 modern basin, clay $\delta^7\text{Li}$ and ϵ_{Nd} values of bank sediments range from -4.1‰ to 0.9‰
7 and from -13.1 to -10.7, respectively. In core CM97, clay $\delta^7\text{Li}$ and ϵ_{Nd} values vary
8 from -2.5‰ to -1.1‰ and from -12.7 to -11.9. We infer that the variations of clay Li
9 isotope compositions are mostly indicative of weathering intensity changes in the
10 Changjiang basin, and are little influenced by mineralogical sorting, lithology and
11 early diagenesis. Over the last 13 kyr, three periods with distinct clay mineralogy and
12 geochemical compositions can be identified. During the Younger Dryas, the positive
13 excursion of clay $\delta^7\text{Li}$ (by $\sim 1.4\text{‰}$) is consistent with enhanced incongruent
14 weathering of silicates (i.e., weakened weathering intensity) in the mid-lower
15 Changjiang basin, likely due to climate cooling. The minor variation of clay $\delta^7\text{Li}$
16 (average $-1.6 \pm 0.2\text{‰}$) at 11 – 2 ka confirms the small changes of weathering
17 conditions, which is consistent with mild climatic variations. Over the last 2 kyr,
18 isotopic fluctuations are best explained by sediment source changes, which might be
19 caused by the intensification of human activities in the mid-lower basin. Overall, this
20 study supports a rapid response of silicate weathering to hydroclimate changes on
21 millennium timescale in large catchments. It also provides new insights on Li isotope
22 fractionations at the continental scale during the Quaternary period.

23

24 **Keywords:** Li and Nd isotopes; clay; Changjiang River; Yangtze; continental

25 weathering; paleoclimate

26 **1. Introduction**

27 Chemical weathering of silicates plays a key role in shaping Earth's surface
28 landscape and modulating biogeochemical cycles (Gaillardet et al., 1999), and it is
29 also one of the most important mechanisms for global climate stabilization (Raymo
30 and Ruddiman, 1992; Frings, 2019). During this process, large amounts of sediments
31 are transported by rivers into marginal seas or deep oceans, and potentially record
32 continental weathering and hydroclimatic variations on various temporal scales. For
33 instance, Lupker et al. (2013) interpreted the Bay of Bengal record as reflecting
34 enhanced weathering intensity and weathering rate since ~21 ka, in response to more
35 favorable Holocene climate than in the Last Glacial Maximum. Similar studies were
36 also carried out worldwide, such as in the Changjiang (Yangtze) Delta and in the Nile
37 Delta (Wang and Yang, 2013; Bi et al., 2017; Bastian et al., 2017).

38 The large river basins cover significant proportions of continental crust and
39 various climatic regimes, and thus give advantages of studying weathering on a global
40 scale (e.g., Gaillardet et al., 1999). However, the complex sediment source-to-sink
41 processes and sedimentary recycling challenge our understanding of both the kinetic
42 and the amplitude of continental weathering response to climatic variability. For
43 instance, the comminution age of fine siliciclastic sediments (<50 μm) in the
44 Changjiang basin can reach 300–400 kyrs (Li et al., 2016). Then, significant time lags
45 may exist in sediment records between weathering proxies and climate variations in
46 large catchments. On the other hand, several lines of evidences have suggested that
47 the weathering-climate feedback observed in sediment records could be ascribed to

48 the changes of sediment sources caused by different erosional patterns within the
49 basin or changes in the sedimentary dynamics during transport (Bi et al., 2017; Wan et
50 al., 2017; Frings, 2019). In spite of these issues, there are examples indicating nearly
51 synchronous co-variations between weathering and paleoclimate variability (Lupker
52 et al., 2013; Bastian et al., 2017). In particular, during specific Quaternary
53 hydroclimate events, negligible changes of clay sources were observed in Nile Delta
54 (Bastian et al., 2017). Overall, the mechanisms and feedbacks linking silicate
55 weathering to climate through time need to be further investigated at large scale.

56 During the last decade, Li isotopes have been considered as one of the most
57 powerful proxies for silicate weathering investigation. Li is predominantly derived
58 from weathering of silicate rocks, even in carbonate dominated catchments (Huh et al.,
59 1998; Kısakürek et al., 2005). Li cation is monovalent and not affected by redox
60 reaction. During weathering processes, primary mineral dissolution shows no
61 significant Li isotope fractionation, which has been verified by laboratory
62 experiments (Pistiner and Henderson, 2003; Wimpenny et al., 2010; Verney-Carron et
63 al., 2011) and by field studies (Ryu et al., 2015). In contrast, secondary minerals
64 preferentially incorporate the light ^6Li , leaving isotopically heavy ^7Li in river water
65 (Huh et al., 1998; Wang et al., 2015). Hence, Li isotope compositions ($\delta^7\text{Li}$) are
66 primarily controlled by the ratio of primary mineral dissolution to the secondary
67 mineral formation. This ratio has been described as weathering congruency or
68 intensity, with congruent weathering (i.e., high ratio) leading to low $\delta^7\text{Li}$, close to the
69 compositions of bedrock (e.g., Misra and Froelich, 2012; Bastian et al., 2017; Pogge

70 von Strandmann and Henderson, 2015; Pogge von Strandmann et al., 2017). Recently,
71 Li isotopes were used to decipher weathering-climate relationship over the geologic
72 past, which suggest distinct response to climate variation. The increase of $\delta^7\text{Li}$ values
73 in the middle-upper Datangpo Formation (Guizhou province, South China) is
74 consistent with an initiation of global cooling before the Marinoan glaciation (Wei et
75 al., 2020). In the Nile basin, Bastian et al. (2017) observed higher clay $\delta^7\text{Li}$ values
76 during several arid periods over the last 30 kyr. In contrast, clay $\delta^7\text{Li}$ displays low
77 values during the last glacial period in sediments from Himalayan terraces (Dosseto et
78 al., 2015). To date, it is still unclear how sediment $\delta^7\text{Li}$, silicate weathering and
79 climate are related over the geologic past.

80 As the largest and longest river in Asia, the Changjiang River originates from the
81 Tibetan Plateau, and flows eastward into the East China Sea. The Changjiang Delta,
82 as one of the major sinks of river sediments, preserves high-resolution sedimentary
83 records for the Holocene environmental changes in this large catchment. In this study,
84 we report clay Li and Nd isotopes and element concentrations in core CM97
85 recovered from the Changjiang Delta and in modern river bank sediments (Fig. 1). In
86 combination with published paleoclimatic records in this region, we investigate how
87 riverine clay Li isotopes may indicate changes in silicate weathering as a function of
88 climatic variations in large river systems.

89

90 **2. Regional background**

91 The Changjiang, as one of the largest rivers in the world, originates from the

92 Tibetan Plateau at an elevation above 5,000 m, and drains central China with a length
93 of ca. 6,300 km and a catchment area of 1.8×10^6 km² (Fig. 1A). The annual average
94 sediment flux was 470 Mt, which represents ca. 2.5% of present-day estimate for the
95 global sediment flux (Milliman and Farnsworth, 2011). Its drainage basin spans
96 three-grade physiographic terraces in China, with average elevations of 3,000 – 5,000
97 m, 500 – 2,000 m and less than 500 m respectively (Fig. 1C). According to
98 hydrological and geographic settings, the Changjiang drainage basin is generally
99 divided into three sections. The upper reaches are the section from river source to City
100 Yichang (Fig. 1B), which are mostly located at the first and second-grade
101 physiographic terraces. Before dam constructions in 2003, the upper reaches
102 contributed at least 70% of the annually sediment flux to the East China Sea (Yang Z
103 et al., 2006). The mid-lower reaches are from City Yichang to the river mouth, mostly
104 <500 m in altitude (Fig. 1B). The floodplain and alluvial plains are well developed,
105 occupying about 44% of the total basin area. The Dongting and Poyang Lakes are two
106 major contributors of sediments into the modern Changjiang mainstream in the
107 middle basin (Fig. 1B).

108 Geologically, the Changjiang drainage basin is primarily located in the South
109 China Block, which is composed of the Yangtze Craton and the Cathaysia Block (Fig.
110 1A). It is bounded by the Qinling-Dabie orogenic belt to the north and by the
111 Songpan-Garze terrain to its northwest. The intermediate, basic (e.g., Emeishan basalt
112 province) and ultra-basic rocks are mainly distributed in the upper basin (Fig. 1B).
113 The Changjiang catchment is strongly affected by the humid sub-tropical Asian

114 monsoon climate, that is warm and humid in summer and cold but arid in winter.
115 Generally, the upper basin is subject to the Indian Summer Monsoon (ISM), while the
116 East Asian Summer Monsoon (EASM) dominates climatic changes in the mid-lower
117 basin. At present, the period from May to October accounts for more than 70% of
118 annual water discharge, with a peak mostly in July. Spatially, the annual precipitation
119 increases eastward, from < 400 mm/yr in east Tibet, to about 1000 mm/yr at
120 Chongqing and > 2,000 mm/yr in lower reaches. The mean annual precipitation over
121 the whole basin is ~1,100 mm, and the water discharge into the sea is about 900
122 km³/yr. Except for the cold climate (below 4°C) in the river source area, the mean
123 annual temperature in the mid-lower basin is about 16 – 18°C.

124 At the last deglaciation, the sea-level in the East China Sea was about 120 m
125 below the present-day position (Fig. 1D), and the modern river mouth was occupied
126 by a huge incised valley at that time, with the width and depth up to 70 km and 80 m,
127 respectively (Li et al., 2002). With the postglacial sea-level rise, the incised valley
128 was gradually filled by the Changjiang-derived sediments. During this period, the
129 annual sediment mass deposited in the Changjiang estuarine and coastal areas was
130 estimated to be 137 – 224 Mt/yr (Wang et al., 2018). With the sea-level reaching its
131 highstand at 7 – 6 ka, the annual sediment influx was about 99 – 113 Mt/yr (Fig. 1D)
132 and the modern delta initiated. During the last 2 kyr, strong human activities caused
133 the annual sediment flux increasing to 162 Mt/yr on average.

134

135 **3. Materials and methods**

136 *3.1. Samples*

137 Core CM97 (~70 m in depth) was drilled at the modern Changjiang River mouth
138 (31°370' N, 121°230' E) in 1997. The AMS ¹⁴C dating on molluscan and/or snail
139 shells was reported by Hori et al. (2001), and then the age model was gained using
140 linear interpolation. Core CM97 thus records the depositional history over the past
141 ~14,000 years, and the sedimentary sequence primarily consists of fluvial and
142 floodplain facies at the bottom (70 – 60.3 m in depth), estuarine to shallow marine
143 facies in the middle part (60.3 – 20.1 m), and deltaic facies at the top (20.1 – 0 m).
144 The detailed information about sedimentary facies was described in Hori et al. (2001).
145 In this study, a total of 61 samples were selected from core CM97 for geochemical
146 measurements. After this, in order to constrain the provenance of core sediment, 11
147 riverine bank samples, including 5 from the upper reaches, 3 from the Dongting and
148 Poyang Lake in the middle basin, and 3 from the lower reaches, were taken in May
149 and July, 2018 (Fig. 1B).

150

151 *3.2. Clay fraction (<2 μm) separation and Li isotope analyses*

152 About 1 g bulk sediment was first separated into two groups (i.e., > 63 μm sand
153 fraction and < 63 μm fine-grained fraction) using wet sieving method. Then, the
154 fine-grained fraction (<63 μm) was immersed into 20 ml 1N HCl for twenty minutes
155 to remove carbonates at room temperature. This method is effective enough for
156 carbonate removal. Carbonate is generally Li-poor (1-2 μg/g), but the use of HCl acid
157 may remove some Li from Fe-Mn oxides/hydroxides (Bi et al., 2017). In this study,

158 we calculate that a maximum of ~6% of sedimentary Li is removed at this step, by
159 comparing Li concentrations before and after HCl leaching. The residue was washed
160 three or four times with Milli-Q water until the supernatant pH reached neutral value.
161 Afterwards, the clay fraction was separated from the fine-grained fraction by the
162 pipette method following the Stoke's Law at room temperature. In order to remove
163 exchangeable Li, the clay fraction was mixed with 20 ml 1 N Ammonia Chloride
164 (NH_4Cl) solution (Vigier et al., 2008). Finally, the clay fraction was freeze-dried after
165 washing twice with Milli-Q water.

166 About 10 mg of clay samples were ground to <200 mesh in an agate mortar and
167 then were digested first using 3:1 HF/ HNO_3 solution at 70°C for several hours.
168 Afterwards, the solution was evaporated and re-dissolved with concentrated HNO_3
169 several times. The sample was then re-digested with 3:1 HCl/ HNO_3 solution. Finally,
170 the solution was evaporated and re-dissolved in 4 ml 1 N HCl. At this stage, the
171 solution was systematically centrifuged to ensure the lack of any solid residue. The Li
172 purification was performed on a cationic resin column, following the method
173 described in Vigier et al. (2008). All of these experiments described above were
174 conducted at Laboratory of Oceanography of Villefranche-sur-Mer (LOV), Sorbonne
175 University. Li isotopes measurement were performed using a MC-ICP-MS Neptune
176 plus (ThermoFisher) at the Ecole Normale Supérieure de Lyon (National Facilities).
177 The accuracy of the isotopic measurements was monitored by analyzing Li7-N pure
178 Li solutions and BE-N basaltic rocks. Repeated measurements yielded mean $\delta^7\text{Li}$
179 value of $+30.3 \pm 0.6\text{‰}$ (2SD, n=26) and $+5.2 \pm 0.5\text{‰}$ (2SD, n=4), respectively. In

180 addition, three samples were processed repeatedly using the same but independent
181 chemical procedure, yielding values of $-1.9 \pm 0.4\text{‰}$ (2SD, n=3), $-1.5 \pm 0.3\text{‰}$ (2SD,
182 n=3) and $-1.3 \pm 0.2\text{‰}$ (2SD, n=3), respectively (Table S2). Thus, the external
183 reproducibility for silicate materials ranges from 0.2‰ to 0.5 ‰ at the 2σ level

184

185 *3.3. Al, Na and Li concentrations and Nd isotope analyses*

186 About 50 mg powder samples were ignited in muffle furnace at 600°C in order to
187 remove organic matter before acid digestion. Afterwards, these powder samples were
188 dissolved with a mixture of 1:1 concentrated HNO₃ and HF in a tightly closed Teflon
189 vessel for at least 48 h with a temperature of 190°C. After drying, samples were
190 re-dissolved in HNO₃ to break down any fluorides. Finally, samples were re-digested
191 in 2 ml 30% HNO₃ with a temperature of 190°C. The concentrations of Al and Na
192 were measured respectively by Inductively Coupled Plasma-Optical Emission
193 Spectrometers (ICP-OES, IRIS Advantage), and of Li by Inductively Coupled Plasma
194 Mass Spectrometry (ICP-MS, Agilent 7900) at the State Key Laboratory of Marine
195 Geology, Tongji University. The analytic precision and accuracy were monitored by
196 rock standards of BCR-2. The results showed that the precision and accuracy of
197 elements is generally less than 5%, while Li concentration has an accuracy of 10%.

198 The Nd purification was conducted with a two-step method. The Rare Earth
199 Elements (REE) were first separated using 2.5 N HCl elution on an ion-exchange
200 column filled with Bio-Rad AG50W-X12, 200-400 mesh resin. Then, the Nd was
201 separated from the REE solution using 0.25 N HCl. Finally, the Nd isotope ratios

202 were determined using a MC-ICP-MS Neptune plus at the State Key Laboratory of
203 Marine Geology, Tongji University. In this study, Nd isotope values are reported as
204 ϵ_{Nd} , which is equal to $[(^{143}\text{Nd}/^{144}\text{Nd})_{\text{samples}}/(^{143}\text{Nd}/^{144}\text{Nd})_{\text{CHUR}}-1] \times 10^4$. The CHUR
205 represents Chondritic Uniform Reservoir, with a value of 0.512638. The mean
206 measured ϵ_{Nd} of BCR-2 is 0.0 ± 0.3 (2SD, n=10), well within the recommended
207 values.

208

209 **4. Results**

210 The Li concentrations of clay fractions from CM97 sediments range from 78.6
211 $\mu\text{g/g}$ to 99.1 $\mu\text{g/g}$, with an average of $88.0 \pm 5.4 \mu\text{g/g}$ (Table 1). Their $\delta^7\text{Li}$ values vary
212 between -2.6‰ and -1.1‰ (Table 1), systematically lower than the average of $+0.6 \pm$
213 0.6‰ estimated for the upper continental crust (Sauzéat et al., 2015). As for the
214 modern bank sediments, clay $\delta^7\text{Li}$ of upstream-derived sediments vary from -0.3‰ to
215 0.9‰ (Table 2). These values are comparable with those reported for sedimentary
216 rocks in the Tibetan Plateau (Weynell et al., 2017), and are within the range for the
217 upper continental crust (Fig. 2A). In comparison, the Li isotope compositions of
218 clay-size sediments collected in the Dongting and Poyang basins are lighter, ranging
219 from -4.1‰ to -2.3‰ (Fig. 2A). As already demonstrated by Wang et al. (2015), the
220 $\delta^7\text{Li}$ values of SPM samples collected in the upper Changjiang reaches are also higher
221 than those in Dongting and Poyang Lakes (Fig. 2A). The ϵ_{Nd} values of clay fractions
222 separated from core CM97 sediments show a narrow but significant variation, ranging
223 from -12.8 to -11.6 (Fig. 2B). This is within the range measured for modern

224 Changjiang river sediments (Yang et al., 2007; He et al., 2015). Clay ϵ_{Nd} values vary
225 from -11.5 to -10.7 in the upper reaches, and from -15.2 to -12.4 in the Dongting and
226 Poyang Lake regions (Table 2). Clay ϵ_{Nd} values in the lower reaches range from -12.2
227 to -11.7. The difference in clay ϵ_{Nd} between the upper reaches and the middle basin is
228 larger than the analytical uncertainty (0.3, 2SD), making possible to use this proxy for
229 assessing the sediment provenance.

230

231 **5. Discussion**

232 *5.1. Clay provenance and depositional environments*

233 The provenance of core CM97 sediments has already been estimated using clay
234 mineral assemblages and bulk sediment Sr-Nd isotope compositions (Wang and Yang,
235 2013; Bi et al., 2017). These results indicated that sediments accumulated in the
236 Changjiang Estuary typically represented an average composition of particulates from
237 the whole Changjiang basin (mainly from the mountainous upper reaches, as well as
238 from the middle basin). Nd isotopes are thought to be independent of Earth surface
239 processes (e.g., chemical weathering and hydrodynamic sorting), and thus are widely
240 used to discriminate river sediment sources (e.g., Bayon et al., 2015; Revel et al.,
241 2015). Given that the Changjiang basin is tectonically stable since the late Pleistocene
242 (Yang S et al., 2006), the provenance of core CM97 clay fraction can be determined
243 by comparing their Nd isotope compositions with those of modern river sediments.
244 The Permian basalts widely distributed in the upper Changjiang basin are
245 characterized by relatively higher ϵ_{Nd} values, compared to those of granites and

246 Quaternary fluvial-lacustrine sediments in the mid-lower basin (Yang et al., 2007; He
247 et al., 2015). Accordingly, the ϵ_{Nd} values of the Changjiang sediments decrease
248 downstream with increasing sediment contribution from the mid-lower basin (Yang et
249 al., 2007). As shown in Figure 3B, the content ratios of kaolinite to illite (i.e.,
250 kaolinite/illite) are also shown for comparison (Wang and Yang, 2013). At present,
251 illite accounts for 60 – 80% of clay mineral assemblages in the upper Changjiang
252 basin, while sediments from the Dongting and Poyang regions in the middle basin are
253 featured by relatively higher kaolinite contents, in relation with more intensive
254 weathering conditions therein (He et al., 2013). Thus, at a first approximation,
255 kaolinite/illite ratios indicate a source effect (i.e., sediment from the upper basin or the
256 middle basin). Indeed, warm and humid climate favors kaolinite formation.
257 Considering the climatic evolution over the past 14 kyr, we therefore suggest that the
258 kaolinite/illite ratio in core CM97 can be affected by changes of silicate weathering in
259 the Changjiang basin as well.

260 As illustrated in Figure 3A, clay ϵ_{Nd} values show a limited variation at 13 – 2 ka
261 and a negative shift during the last 2 kyr. It is worth noting that the negative
262 excursions of clay ϵ_{Nd} at 4.3 – 4.1 ka are small but larger than analytical uncertainty,
263 suggesting more clay contributions from the middle basin during this period. This
264 result is consistent with the observation from kaolinite/illite ratio, which shows stable
265 values at 11 – 2 ka but higher values at 2 – 0 ka (Fig. 3B). This consistency between
266 clay ϵ_{Nd} and kaolinite/illite ratios evidence a significant change of the sediment source
267 during the last 2 kyr, with a greater but fluctuating contribution from the middle

268 Changjiang basin. In contrast, at 13 – 11 ka (i.e., Younger Dryas period),
269 kaolinite/illite ratio displays a slightly decreasing trend, while clay ϵ_{Nd} variation
270 remains stable. This may feature a progressive shift of chemical weathering
271 conditions during the Younger Dryas, which we will be investigated further using Li
272 isotopes (section 5.3).

273 With the sea-level rise during the last deglaciation, the local depositional
274 environment has greatly changed in the Changjiang Delta. The sedimentary system in
275 the East China Sea shifted from the transgressive system tract in the last deglaciation
276 (15.4 – 7 ka) to the highstand system tract since 7 ka (Hori et al., 2001; Li et al., 2002).
277 Correspondingly, in the core CM97, the fluvial facies, estuarine to shallow marine
278 facies, deltaic facies and their respective sub-facies can be subsequently observed (Fig.
279 1E). The changes in depositional environment may significantly affect provenance
280 discrimination and weathering information recorded by core sediments. For instance,
281 as shown in Figure 3C, 3D and 3E, bulk ϵ_{Nd} values are higher in coarser sediments at
282 periods with higher sedimentation rates (i.e., at 13 – 11 ka and 2 – 0 ka). In contrast,
283 clay ϵ_{Nd} values appear independent from the variation of mean grain size and
284 sedimentation rate. Hence, there is an apparent inconsistency for sediment source
285 between clay fraction and bulk sediments in core CM97. This inconsistency is best
286 explained by a different transport and deposition between coarse-grained sediments
287 and clays. Fine-grained clays are transported more rapidly and likely less sensitive to
288 these physical processes. These observations strongly suggest that geochemical
289 compositions of bulk sediments can be largely affected by depositional environment

290 and sea-level changes, while fine clays remain less sensitive to these aspects, as
291 suggested by study in the Nile Delta (Bastian et al., 2017).

292

293 *5.2. Grain size, lithological, estuarine and diagenetic effects on clay $\delta^7\text{Li}$*

294 In this study, in order to minimize grain size effect, we focused on clay fractions.
295 Li is generally more concentrated in clay minerals, and coarse-grained quartz and
296 feldspar are Li-poor host (Dellinger et al., 2014; Sauzéat et al., 2015). Indeed, Li and
297 $\delta^7\text{Li}$ in riverine sediments, even in fine suspended particulate matter, can be strongly
298 influenced by a mixing between various primary minerals and weathering products
299 (e.g., Dellinger et al., 2014, 2017). The Changjiang basin is characterized by igneous
300 and sedimentary rocks, while high-grade metasedimentary rocks are distributed
301 sporadically (Yang et al., 2007). As illustrated in Figure 4A, the geochemical
302 compositions of SPM samples and bulk sediments of core CM97 both show a binary
303 mixing between igneous end-member and weathering products. In contrast, $\delta^7\text{Li}$
304 values and Li concentrations measured in clay fractions are clearly distinct from this
305 mixing pattern (Fig. 4). This implies that elemental and isotopic variations in clay
306 samples are not derived from mixing with igneous rocks. Compared to the bulk
307 sediments, the clay fraction and fine-grained sediments in general (as also shown in
308 Dellinger et al., 2017) are more likely to contain a higher proportion of
309 contemporaneous weathering products (i.e., soil-derived clays). This observation
310 therefore strongly supports the use of clay fraction as a tracer of continental
311 weathering. However, the trend defined by clay fraction also demonstrates some

312 potential influences from recycled sedimentary rocks, i.e. recycled clays derived from
313 sedimentary rocks (Fig. 4). In the Changjiang basin, illite is the most abundant clay
314 minerals in upstream sediments (He et al., 2013), and is mostly eroded from recycled
315 sedimentary rocks (Weynell et al., 2017). Kaolinite is a weathering product that is
316 mainly produced in the middle Changjiang basins (He et al., 2013). To some extent,
317 the influence of recycled sedimentary rocks on chemical weathering can thus be
318 roughly estimated by kaolinite/illite ratios.

319 Estuarine processes may also affect the geochemical compositions of deposited
320 particles and sediments. Due to the difference in ionic strength, pH and temperature
321 between river and sea water, chemical compositions of riverine sediments can be
322 modified in the estuary by adsorption-desorption, particle dissolution and/or ongoing
323 weathering processes (Gislason et al., 2006; Jeandel and Oelkers, 2015). For Li
324 isotopes, there is no consensus about the influence of these processes, and only few
325 studies have been published. In Iceland, Pogge von Strandmann et al. (2008) observed
326 an increase of SPM $\delta^7\text{Li}$ values (from $\sim 0\%$ to $\sim 5\%$) along a transect in the
327 Borgarfjörður estuary, and related this to ongoing weathering of particles with
328 seawater. Based on the investigation of sedimentary facies, the location of core CM97
329 was suggested to be inundated since $\sim 9 - 10$ ka due to sea-level rise (Hori et al., 2001;
330 Li et al., 2002). However, clay $\delta^7\text{Li}$ values fluctuate around -1.5% during the middle
331 Holocene under the maximum marine transgression (Fig. 5A), and show no positive
332 or negative excursion in response to ongoing reaction with seawater or particle
333 dissolution. Additionally, any adsorbed Li onto clay or Fe-Mn oxyhydroxides surface

334 has been removed using NH_4Cl solution (see section 3.2). Thus, estuarine processes
335 did not visibly alter clay $\delta^7\text{Li}$ values over the studied period of time.

336 Early diagenesis is another process that may be associated to the uptake of Li
337 from seawater ($\delta^7\text{Li} = 31.2\text{‰}$), with a non-negligible isotope fractionation (Misra and
338 Froelich, 2012). Clay $\delta^7\text{Li}$ values of core CM97 should vary with the proceeding of
339 diagenesis, as authigenic clays generated from seawater are expected to result in
340 higher $\delta^7\text{Li}$ values relative to continental weathering products (Wei et al., 2020).
341 However, there is no systematic increase or change of the clay $\delta^7\text{Li}$ value as a
342 function of depth or of the sediment age (Fig. 5A). Also, in Figure 4, most clay data
343 plot close to the shale end-member (Fig. 4). These observations therefore strongly
344 suggest that early diagenesis was not significant enough to alter the clay $\delta^7\text{Li}$ values
345 inherited from continental process. In fact, these relatively young detrital clays
346 contain large amounts of lithium, which may mask the signal from minor lithium
347 addition or loss potentially associated with early diagenesis.

348

349 *5.3. Assessing links between weathering and climatic variation*

350 As illustrated in Figure 5A, core CM97 clay $\delta^7\text{Li}$ values exhibit a small but
351 statistically significant increase of $\sim 1.4\text{‰}$ during the Younger Dryas (YD) from 13 ka
352 to 11 ka. Then, relatively stable $\delta^7\text{Li}$ values (compared to the external reproducibility)
353 are punctuated by excursions at three short-term periods between 11 ka and 2 ka.
354 These excursions occurred at 4.2 – 4.1 ka, 8.6 – 8.1 ka, 10.5 – 10.1 ka, possibly
355 corresponding to the 4.2 ka, the 8.2 ka, the 10.3 ka climate events (Wanner et al.,

356 2011). However, the ^{14}C ages used in this study may bear large uncertainties, and the
357 temporal resolution is not sufficient to confirm if these fluctuations correspond to
358 these specific climate events. Thus, the mechanism for clay $\delta^7\text{Li}$ variations on
359 centennial timescales will not be further discussed in the following. During the last 2
360 kyr, larger fluctuations of clay $\delta^7\text{Li}$ (between -1.1‰ and -2.5‰) can be observed
361 (Table 1).

362

363 *5.3.1. Impact of climate cooling during the Younger Dryas*

364 The YD event corresponds to millennium-scale climate cooling, and punctuated
365 the termination of the last glacial period at 12.9 – 11.7 ka. During this period, the
366 temperature in central China was estimated to be $\sim 2 - 4^\circ\text{C}$ lower, based on branched
367 fatty alcohol ratio (BNA15) measured in Dajiuhu peatland (Fig. 5E; Huang et al.,
368 2013). According to paleoclimatic records recovered from Tiancai lacustrine
369 sediments (Zhang et al., 2017), the temperature decrease was also observed in the
370 upper Changjiang basin (Fig. 5D).

371 As a first approximation, the positive excursion of clay $\delta^7\text{Li}$ during the YD
372 period could be caused by a change of Li isotope fractionation factor, as it is
373 temperature-dependent (Vigier et al., 2008). However, a temperature decrease would
374 rather lead to a decrease in clay $\delta^7\text{Li}$ values (Vigier et al., 2008). Then, a change of
375 sediment source can be envisioned, but this is not consistent with the minor changes
376 of clay ϵ_{Nd} values during this period (Fig. 5C). We therefore propose that the variation
377 in core CM97clay $\delta^7\text{Li}$ during the YD period is most likely indicative of changes in

378 silicate weathering intensity in response to climatic evolution. As Li is quantitatively
379 incorporated into the secondary minerals, clay $\delta^7\text{Li}$ values primarily depend on the
380 ratios of primary mineral dissolution to secondary minerals formation. Higher
381 temperature is expected to increase the congruency of weathering, as the solubility of
382 most primary minerals increases with temperature (Pogge von Strandmann et al.,
383 2017). Conversely, the climate cooling during the YD period appears consistent with
384 more incongruent weathering conditions (i.e., weakened weathering intensity),
385 resulting in higher clay $\delta^7\text{Li}$ values. The decreasing trend of kaolinite/illite ratio
386 during the YD period (Fig. 5B) supports this interpretation, since less kaolinite
387 formed in the middle Changjiang basin is consistent with a cool climate.

388

389 *5.3.2. Impact of Holocene monsoon variability between 11 ka and 2 ka*

390 As mentioned above (Fig. 1A), the upper Changjiang basin is subject to the India
391 Summer Monsoon (ISM), while the climate in the mid-lower basin is controlled by
392 the East Asia Summer Monsoon (EASM). The variation of ISM is directly linked to
393 solar insolation. In contrast, apart from solar insolation, ice volume in North Atlantic
394 and the West Pacific subtropical high possibly exert additional forcing on the EASM
395 (Dykoski et al., 2005, Rao et al., 2016). Consequently, the ISM and EASM show
396 synchronous variations during the Holocene in the Changjiang basin (Figs. 5D and
397 5E).

398 Between 11 ka and 2 ka, clay ϵ_{Nd} variations are small compared to analytical
399 uncertainty (Fig. 3A), which implies relatively stable source of core CM97 clay

400 fraction. The kaolinite/illite ratios and clay $\delta^7\text{Li}$ variations remain minor as well (Figs,
401 5A and 5B). Previous studies have indicated that the response of weathering intensity
402 to climatic variation can be dampened in large river catchments (Li et al., 2016;
403 Romans et al., 2016). Indeed, due to the long residence time of sediments, large river
404 basins can buffer any trace of changes caused by climatic variations (Li et al., 2016;
405 Romans et al., 2016). However, this view is not consistent with the significant
406 variations of Li isotope observed during the YD period (Fig. 5A). Clays being the
407 finest fraction in river sediments may be transported rapidly and reside over a much
408 shorter timescale in the flood/alluvial plains, such as in the Nile Basin (Bastian et al.,
409 2017). Thus, in the context of negligible sediment source changes, the minor changes
410 of clay $\delta^7\text{Li}$ in core CM97 demonstrate insignificant variation of weathering intensity
411 in the Changjiang basin at 11 – 2 ka. As illustrated in Figures 5D and 5E, climate
412 variations in the Changjiang basin were mild during this period, which may explain
413 this minor variation of continental weathering.

414

415 *5.3.3. Impact of human activities during the last 2 kyr*

416 During the last 2 kyr, clay ϵ_{Nd} and kaolinite/illite ratios both indicate additional
417 clay contribution from the middle Changjiang basin as discussed in section 5.1. Thus,
418 changes in clay source most likely account for the observed variation of clay $\delta^7\text{Li}$ in
419 core CM97 (Fig. 5A). Over the last 2 kyr, a fast development of society and
420 civilization is observed in the Changjiang basin (Wang and Yang, 2013; Bi et al.,
421 2017). For instance, the reduction in trees and shrubs pollen, in parallel with a rise of

422 herbaceous pollen, suggest enhanced agricultural activities in the lower Changjiang
423 basin since 3 ka (e.g., Yao et al., 2017). In the past 1,000 years, the lower Changjiang
424 basin became a center of intensive rice production (Zong et al., 2012). Soil erosion
425 can be strongly affected by human activities, especially by cultivation and
426 deforestation (Wang et al., 2011). Accordingly, relative to the period between 11 ka
427 and 2 ka, more kaolinite could be eroded from the middle basin and transported
428 further to the Changjiang Estuary. Nevertheless, the analytical uncertainty of the ^{14}C
429 dating and sampling resolution in this study do not allow us to further investigate the
430 mechanism of how human activities altered the clay sediment production and
431 transport. More direct evidences and high-resolution observations are further required
432 for a better understanding of the anthropogenic perturbation on weathering and
433 erosion processes in large catchments.

434

435 **6. Implications and Conclusions**

436 The glacial-interglacial cycle is of particularly importance to understand the
437 weathering-climate relationships (Frings, 2019). However, hydrodynamic sorting,
438 sediment recycling and long sediment residence time in the flood/alluvial plains make
439 the interpretation of weathering proxies ambiguous, in particular for a large river
440 basin. In this study, the Changjiang Delta provides high-resolution sediment archives
441 for investigating weathering and climatic variation over the past 14, 000 years. Our
442 results indicate that climate remains a major external force for silicate weathering at
443 the continental scale. They also underline the importance to establish changes in the

444 sediment source in response to hydroclimate variations.

445 We observe that clay Li isotope compositions faithfully record the YD event,
446 while source tracers (e.g., Nd isotopes) remain constant. This strongly suggests a fast
447 response of chemical weathering to a strong climate event in the Changjiang basin.
448 The weathering signals registered by the clay fractions are less altered during
449 sediment “source-to-sink” processes because of their relatively rapid transport by
450 waters and negligible mineral sorting effect. Possibly, the comminution age calculated
451 by Li et al. (2016) for fine-grained sediments (<50 μm) in the Changjiang basin
452 concern mostly the coarser minerals (>5 – 10 μm).

453 Another interesting feature is that clay $\delta^7\text{Li}$ and ϵ_{Nd} values fluctuate
454 significantly during the last 2 kyr, suggesting a more intensive human-induced soil
455 erosion. The minor $\delta^7\text{Li}$ and ϵ_{Nd} variations at 11 – 2 ka are likely caused by small
456 changes of weathering conditions, in agreement with mild climatic variations during
457 the Holocene in the Changjiang basin. Overall, the clay $\delta^7\text{Li}$ records suggest the
458 complex response of silicate weathering and clay formation in large catchments on
459 various temporal scales.

460

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639

Table 1 Concentrations of Al, Na and Li and Li-Nd isotope compositions of core CM97 clay fraction

Sample ID	Age cal. yr BP	Al wt%	Na wt%	Li μg/g	clay $\delta^7\text{Li}$ ‰	clay ϵ_{Nd}
CM2.4	176	6.3	0.2	83.2	-2.5	-12.1
CM4	293	9.8	0.2	92.8	-1.3	-12.2
CM6.05	444	9.4	0.4	79.7	-1.4	-12.6
CM8.3	623	8.2	0.4	83.3	-1.7	-12.3
CM10.5	770	9.8	0.3	78.8	-1.3	-12.7
CM12.6	924	11.7	0.5	81.5	-1.6	
CM14	1027	8.1	0.4	79.5	-1.6	-12.6
CM16.3	1210	8.2	0.4	80.7	-1.9	-12.2
CM17.5	1283	11.4	0.2	86.3	-1.3	-12.8
CM18.5	1356	11.9	0.3	90.1	-1.5	
CM19.6	1437	9.2	0.3	86.4	-1.1	-12.3
CM20.7	1518	10.7	0.3	92.8	-1.2	-12.1
CM20.88	1762	10.0	0.3	88.0	-1.4	-11.9
CM20.94	1859	11.0	0.3	92.5	-1.4	-11.9
CM21.13	2165	8.4	0.2	81.7	-1.3	-11.8
CM21.23	2326	10.6	0.3	92.0	-1.6	-11.8
CM21.4	2600	10.8	0.2	90.2	-1.4	-11.7
CM21.68	3051	9.9	0.3	87.8	-1.3	-11.9
CM21.9	3406	9.4	0.3	88.8	-1.2	-12.1
CM22.07	3680	10.2	0.3	92.4		-11.8
CM22.15	3809	9.4	0.3	90.9	-1.6	-11.9
CM22.33	4099	8.2	0.3	80.3	-1.6	-11.6
CM22.37	4164	8.4	0.3	86.5	-1.1	-11.9
CM22.4	4212	9.9	0.3	94.4	-1.9	-12.3
CM22.49	4357	9.6	0.3	90.7	-1.6	-11.7
CM22.66	4621	10.3	0.3	94.6		-11.6
CM23	4816	8.5	0.3	91.2	-1.4	-12.0
CM23.7	5219	12.5	0.3	90.5	-1.4	-12.0
CM24.6	5838	8.4	0.3	95.6	-1.5	-12.1
CM24.95	6256	11.7	0.3	87.8	-1.5	-12.2
CM25.3	6689	10.5	0.3	95.6	-1.7	-11.9
CM25.5	6888	7.1	0.4	93.4	-1.4	-12.1
CM26.1	7211	9.0	0.3	97.8	-1.5	-12.1
CM26.7	7535	10.9	0.3	92.6	-1.5	-12.2
CM27.2	7805	9.5	0.2	86.4	-1.4	-12.0
CM27.54	8042	11.6	0.2	85.3	-1.5	-11.7
CM27.65	8132	8.9	0.2	80.6	-1.9	-12.0
CM27.78	8237	9.1	0.2	86.6	-1.1	-11.8
CM27.9	8335	8.3	0.3	82.6	-1.3	-12.1
CM28.3	8660	9.2	0.3	84.6	-2.0	-11.7

CM28.6	8903	10.0	0.3	92.9	-1.7	-12.2
CM29.4	9283	8.4	0.2	85.9	-1.7	-11.9
CM30.6	9466	9.3	0.2	93.5	-1.8	-12.0
CM32.3	9725	10.6	0.2	84.4	-1.6	-11.9
CM33.4	9893	10.3	0.3	94.4	-1.8	-12.0
CM35	10137	12.0	0.3	89.3	-2.2	-12.0
CM35.5	10213	9.0	0.3	78.6	-1.6	-11.9
CM36.3	10335	11.7	0.3	87.7	-1.1	-11.8
CM36.85	10419	8.7	0.3	80.9	-1.7	-12.1
CM37.5	10518	10.3	0.3	83.0	-1.9	-11.7
CM40.2	10930	9.1	0.4	84.2	-1.6	-11.9
CM42.6	11168	7.9	0.3	87.4	-1.8	-12.0
CM45.2	11426	9.0	0.3	82.9	-1.3	-12.0
CM48.3	11734	9.9	0.3	94.1	-1.2	-12.2
CM50.8	11982	8.9	0.2	96.5	-1.2	-12.0
CM53.7	12270	9.1	0.3	85.3	-1.7	-12.1
CM57.2	12617	9.3	0.3	88.1	-1.7	-12.0
CM59.3	12825	10.2	0.3	94.5	-1.9	-12.2
CM61.45	13039	12.5	0.4	91.7	-2.6	
CM64.1	13302	11.5	0.3	99.1	-2.2	
CM69.5	13838	11.6	0.5	79.9	-1.5	

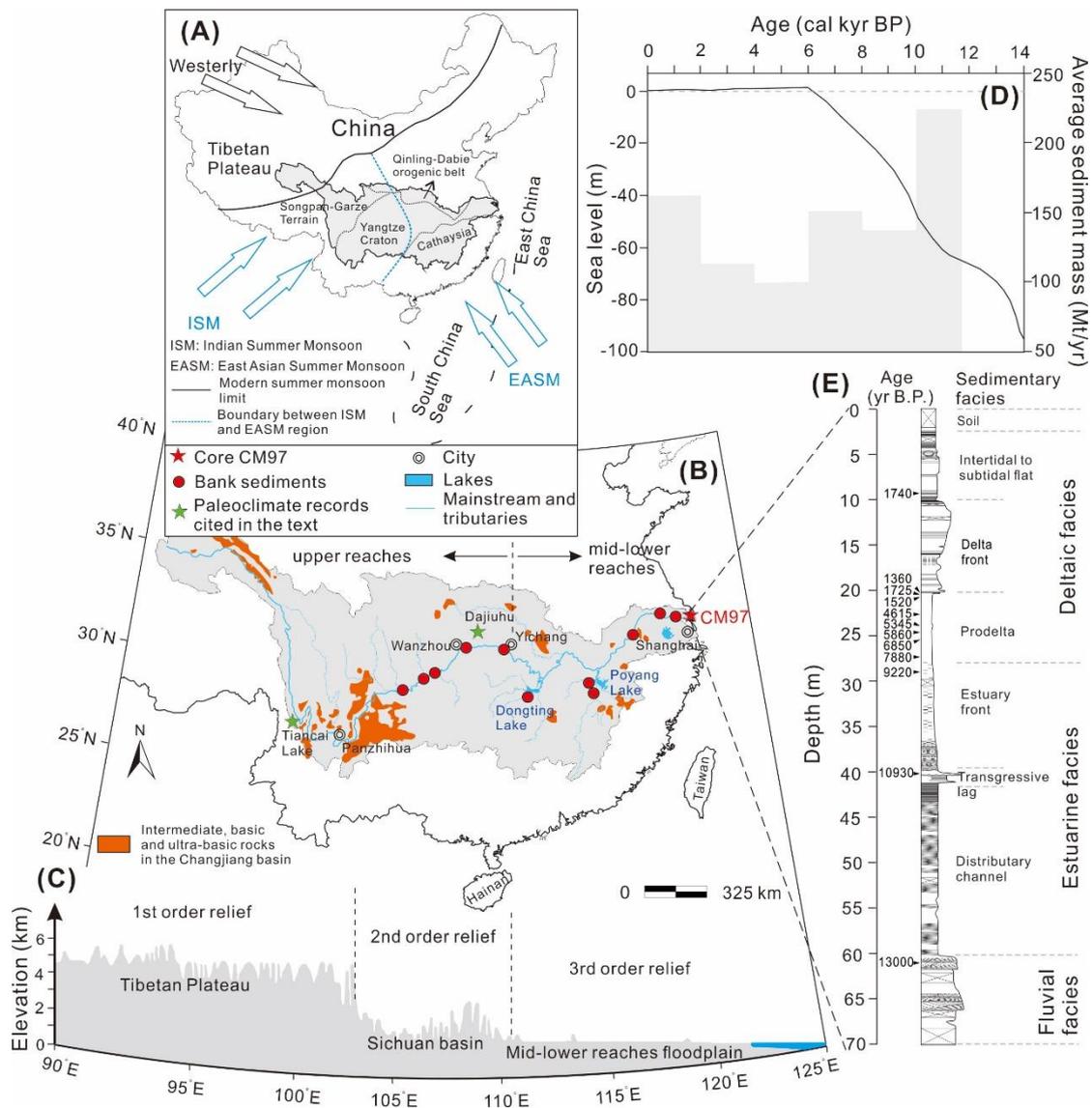
640

Table 2 Li-Nd isotope compositions of clay fraction in bank sediments collected from the modern Changjiang catchment

Sample ID	Location	Longitude °E	Latitude °N	clay $\delta^7\text{Li}$ ‰	clay ϵ_{Nd}
CQ		106.5196	29.4228	0.3	-11.5
HJ		105.8258	28.8248	0.9	-11.3
LZ	Upper reaches	105.3709	28.8546	-0.3	-10.9
WZ		108.4267	30.7615	0.5	-11.5
YZ18YC		111.2742	30.7047	0.5	-10.7
YJCD18		111.5201	28.9361	-2.3	-13.1
YTXJ18	Middle basin	116.8014	28.4763	-3.9	-12.3
NCGJ		116.0165	28.8342	-4.1	-12.4
YZ18AQ		116.9567	30.4478	-1.1	-12.2
YZ18-ZJ	Lower reaches	119.3108	32.2011	-1.4	-11.7
YZ18NT		120.5689	32.0458	-1.4	-11.8

641

Figure captions



643

644 **Fig. 1.** Schematic map showing (A) Asian monsoon regimes, and major tectonic

645 blocks, (B) sampling sites, drainage basin and (C) topography in the Changjiang basin.

646 (D) The sea level changes since 14 ka in the East China Sea (Hori et al., 2001) and

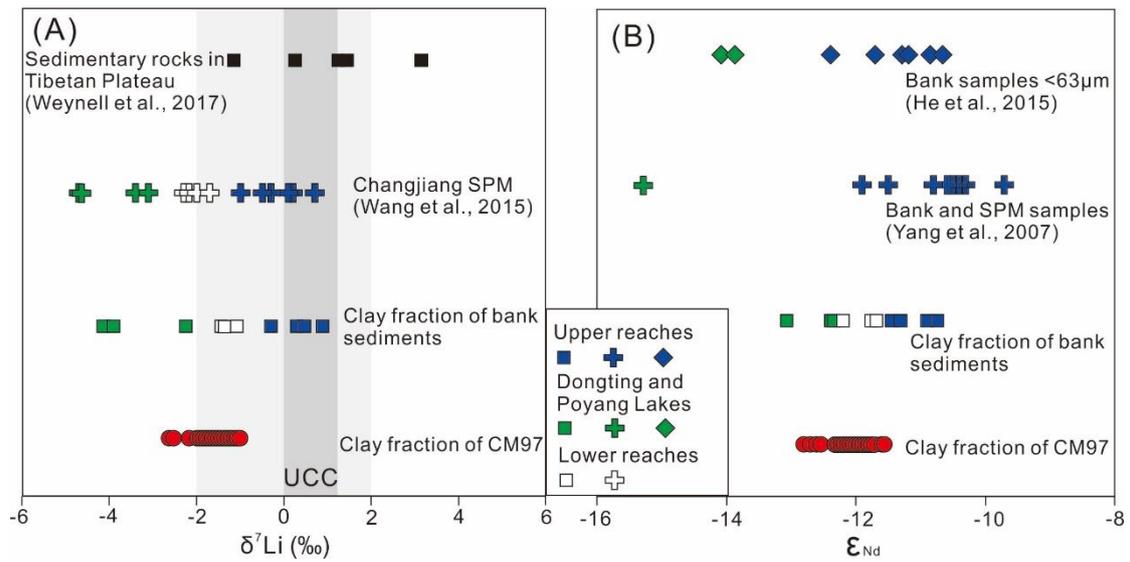
647 annual sediment mass deposited in the Changjiang estuarine and coastal areas (grey

648 bars, Wang et al., 2018). (E) Downcore variation of sedimentary facies and

649 depositional ages of CM97. The detailed description about sedimentary facies can

650 refer to Hori et al. (2001).

651



652

653 **Fig. 2.** (A) Li and (B) Nd isotope compositions of the sediments from the modern

654 Changjiang River and from core CM97. $\delta^7\text{Li}$ values of suspended particulate matter

655 (SPM) are from Wang et al. (2015). The $\delta^7\text{Li}$ values of sedimentary rocks (i.e.

656 mudstone, sandstone and shale) collected from northeastern Tibetan Plateau are from

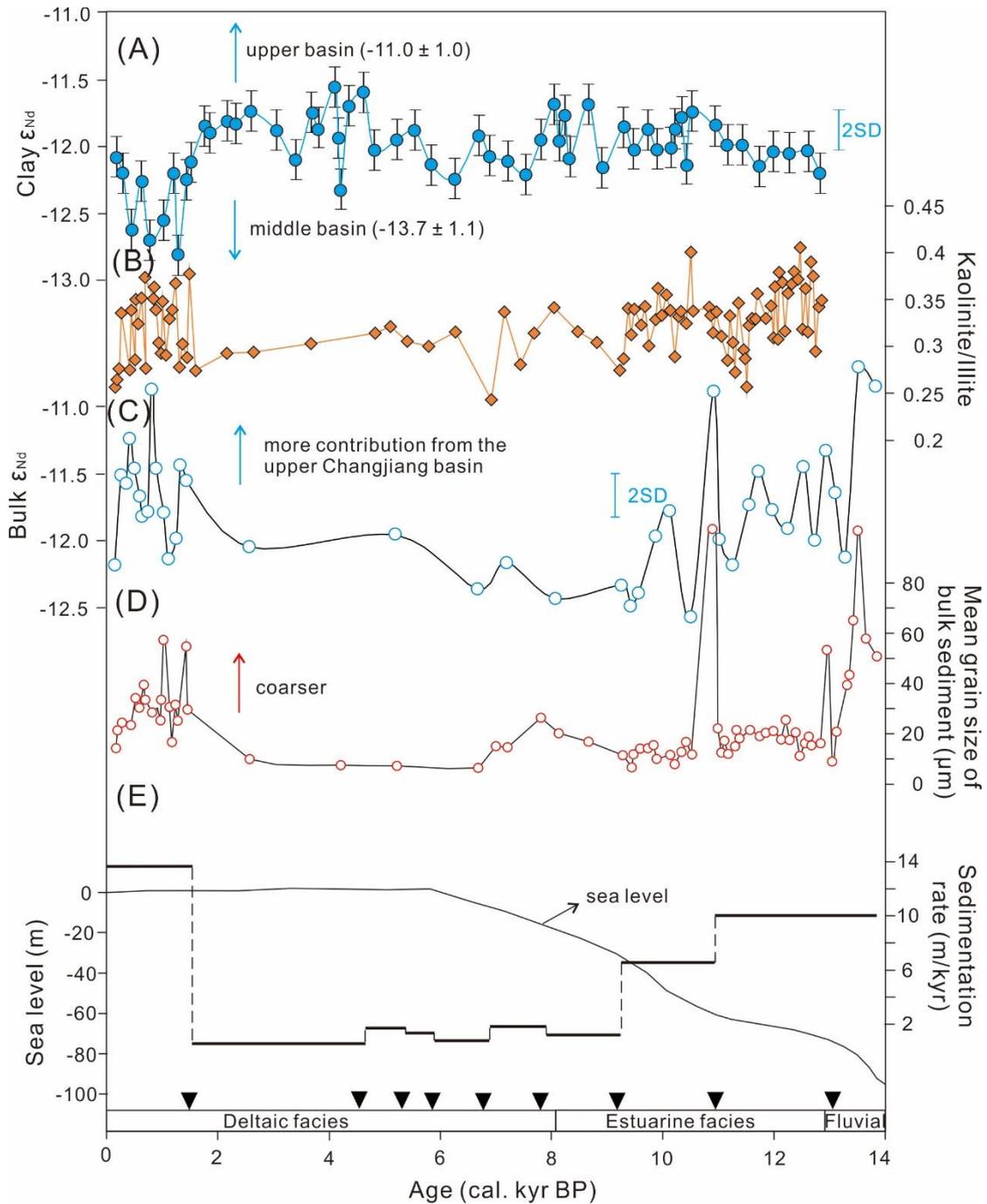
657 Weynell et al. (2017). The light and dark grey bars reflect the average $\delta^7\text{Li}$ values for

658 the upper continental crust (UCC) published by Teng et al. (2004) and Sauzéat et al.

659 (2015), respectively. The Nd isotope compositions of SPM and bank samples are from

660 Yang et al. (2007) and He et al. (2015). The error bar is smaller than symbol size.

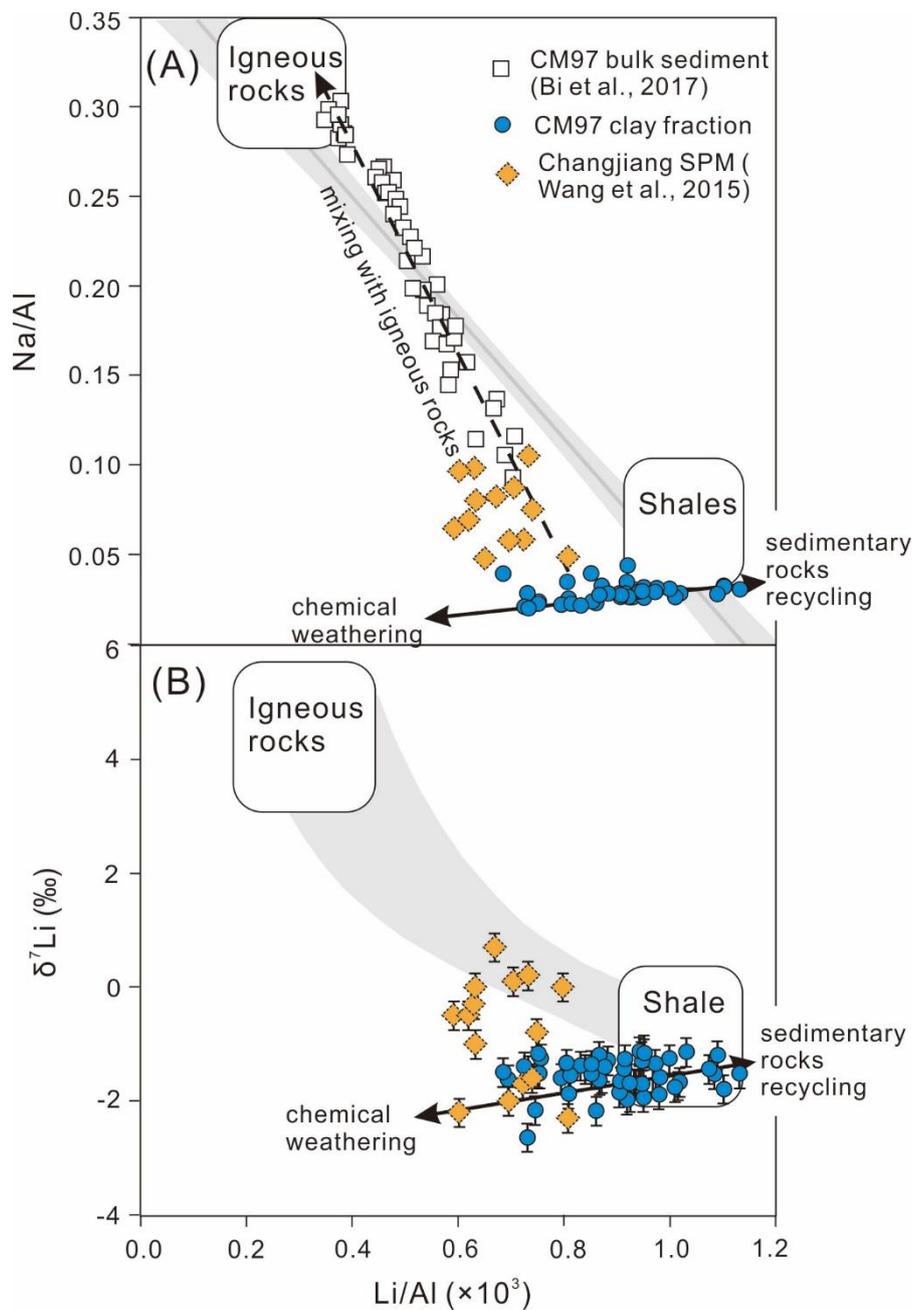
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662

663 **Fig. 3.** (A) Clay ϵ_{Nd} , (B) content ratios of kaolinite to illite
 664 sediments, (C) ϵ_{Nd} values in bulk
 665 sediments, (D) mean grain size of bulk sediment, and (E) mean sedimentation rate for
 666 the core CM97. The error bar for clay Nd isotope is 0.3 (2SD). The postglacial
 667 sea-level changes in the East China Sea are also shown. Sea-level changes and mean
 668 grain size are from Hori et al. (2001) and Bi et al. (2017). Kaolinite and illite contents
 are from Wang and Yang (2013). Bulk Nd isotope compositions are from Bi et al.

669 (2017). The inverted triangles represent sampling positions of molluscan shells which
 670 were used for ^{14}C dating (Hori et al., 2001). The end-member ϵ_{Nd} values of the upper
 671 Changjiang basin, the Dongting and Poyang Lakes are estimated by averaging data
 672 analyzed in this study and those published by Yang et al. (2007) and He et al. (2015).
 673

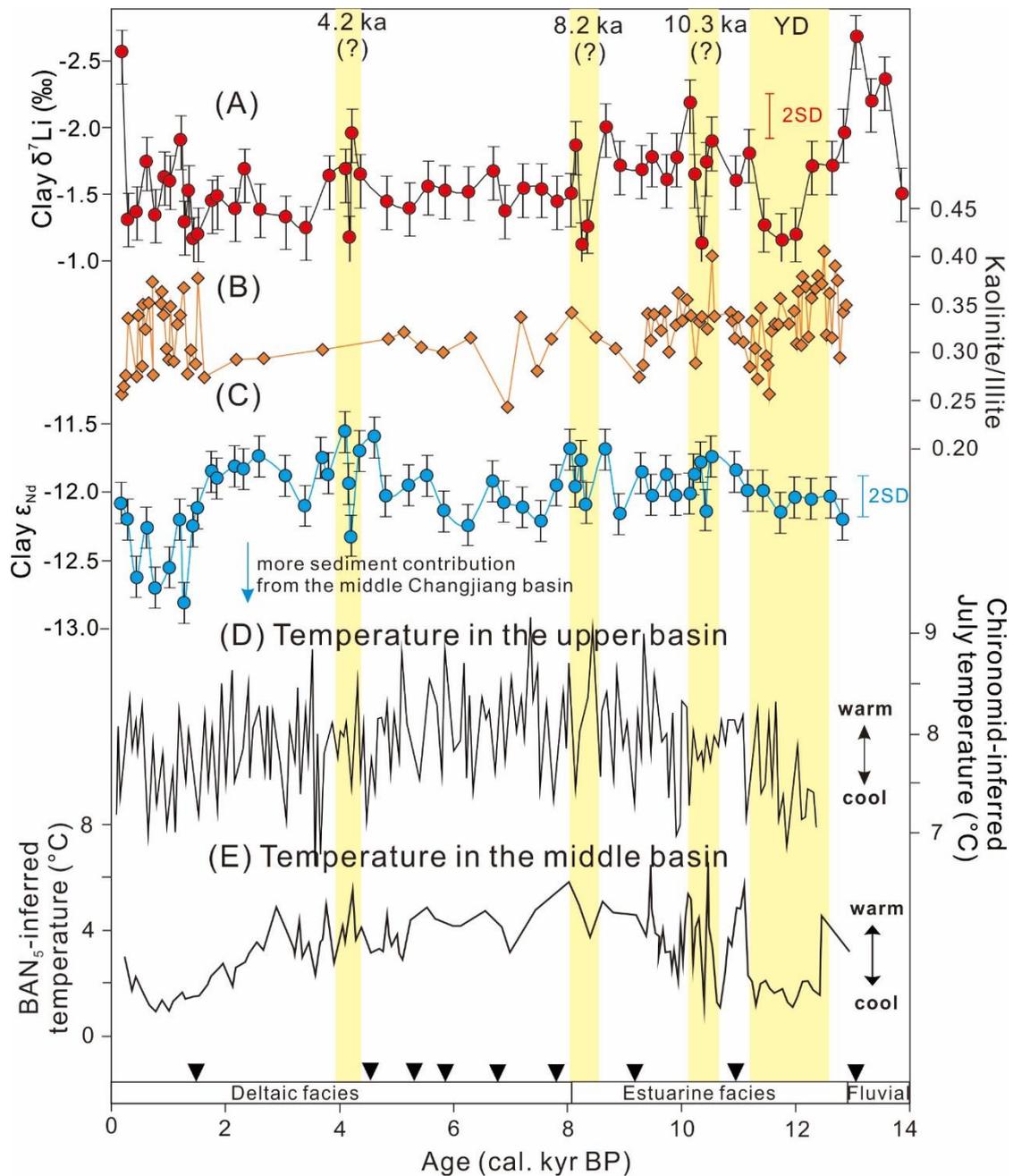


674

675 **Fig. 4.** (A) Na/Al ratios and (B) $\delta^7\text{Li}$ values as a function of Li/Al ($\times 10^3$). The

676 elemental concentrations of CM97 bulk sediments and suspended particulate matter
 677 (SPM) in the Changjiang basin are cited from Bi et al. (2017) and Wang et al. (2015).
 678 The end-members of igneous rocks and shales, and the mixing line with 95%
 679 confidence interval (grey) are cited from Dellinger et al. (2014). The error bar for clay
 680 $\delta^7\text{Li}$ is 0.5‰ (2SD).

681



682

683 **Fig. 5.** Comparison of (A and C) clay Li-Nd isotopes and (B) kaolinite/illite ratios of
684 CM97 with the temperature and rainfall records in the Changjiang basin. (D)
685 Chironomid-inferred mean July temperature in Tiancai Lake, southeastern margin of
686 the Tibetan Plateau (Zhang et al., 2017). (E) Temperature reconstructed by branched
687 fatty alcohol ratio (BNA₁₅) in Dajiuhu peatland (Huang et al., 2013). The error bar for
688 clay $\delta^7\text{Li}$ is 0.5‰ (2SD).