

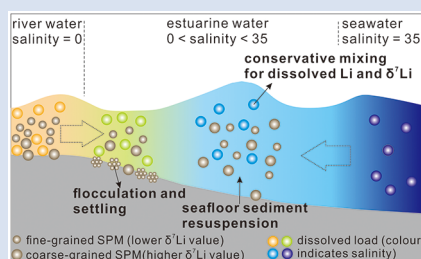
Decoupling of dissolved and particulate Li isotopes during estuarine processes

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Abstract



Lithium isotopes in marine authigenic or detrital sedimentary archives have been recently used to trace continental weathering over geologic timescales. However, interpretations are predominantly based on the assumption that riverine Li isotopic signals can be propagated through estuaries without modification. Here, we verify this hypothesis by investigating the behaviour of Li isotopes in the Changjiang (Yangtze) River estuary. We observe a conservative mixing of dissolved Li and its isotopes between the Changjiang River water and seawater. The dissolved $\delta^7\text{Li}$ yields a non-linear increase with salinity, and a significant increase occurs during the initial water mixing. Through the studied transect, estuarine flocculation and resuspension processes cause the homogenisation of offshore particulate $\delta^7\text{Li}$ values, which are close to the average composition of upper continental crust. This study provides clear and direct evidence that the riverine dissolved Li isotopic signal is not modified during estuarine processes in large rivers, but caution should be exercised when using detrital $\delta^7\text{Li}$ in marginal seas to investigate past continental weathering.

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Introduction

As critical interfaces between continents and oceans, estuaries typically feature gradients of ionic strength and suspended particle concentration, providing an ideal environment for studying land–sea and water–sediment interactions. Over the last decades, numerous studies have demonstrated that riverine elemental fluxes and isotope compositions (*e.g.*, Si, Sr, and Nd) can be modified by physical, chemical, and/or biological processes during their transport through estuaries (Lacan and Jeandel, 2005; Jones *et al.*, 2012, 2014; Zhang *et al.*, 2020). For instance, the dissolution of basaltic particles and Ca–Na exchange on clay minerals in saline water were considered to play non-negligible roles in global climate stabilisation (Gislason *et al.*, 2006; Tipper *et al.*, 2021). Investigating elemental and isotopic alteration of particles and water in estuaries is thus essential for a better understanding of oceanic elemental cycles, and of the carbon cycle in particular.

River Li isotopes ($\delta^7\text{Li}$) are thought to be a powerful proxy of continental weathering (Huh *et al.*, 1998; Wang *et al.*, 2015; Dellinger *et al.*, 2017). During weathering processes, the light ^6Li isotope is preferentially incorporated into the solids, causing the dissolved phase to be isotopically heavy. To date, Li isotopes registered in bulk carbonate or detrital sedimentary archives have been widely applied to assess changes of continental weathering regimes during mass extinctions and long or short term global warming/cooling events (Misra and

Froelich, 2012; Pogge von Strandmann *et al.*, 2013; Bastian *et al.*, 2017; Yang *et al.*, 2021). All these studies implicitly or explicitly assume that riverine Li fluxes to the ocean and their isotopic signals behave conservatively in estuaries. However, thus far, this assumption has not been verified over large scales. Indeed, few case studies have highlighted either the conservative or non-conservative behaviour of Li isotopes in small estuaries (Pogge von Strandmann *et al.*, 2008; Murphy *et al.*, 2014). For instance, Pogge von Strandmann *et al.* (2008) observed an increase of particulate $\delta^7\text{Li}$ values (from ~0 ‰ to ~5 ‰) along a transect in the Borgarfjörður estuary (Iceland) related to ongoing weathering of suspended particles.

Major rivers in Asia, such as the Changjiang (Yangtze) River (Fig. S-1a), drain large continental basins and deliver huge amounts of detrital particles, dissolved elements, and nutrients to the marginal seas and oceans. Detailed investigations of elemental geochemical behaviours in these estuaries provide constraints on the application of Li isotopes as a robust weathering proxy. We present Li isotopic data for the dissolved load, suspended particulate matter (SPM), and their exchangeable phase (see Supplementary Information) in the Changjiang River estuary (Fig. S-1). Our primary goal is to investigate the dissolved and particulate Li isotopic compositions during the mixing processes, and to evaluate the propagation and alteration of terrestrial Li isotopic signals in a large, turbid, and highly dynamic river estuary.

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Evidence for Conservative Mixing of Dissolved Li and $\delta^7\text{Li}$

As shown in Figure 1a, the dissolved Li concentrations are linearly correlated with salinity, suggesting that the Li-poor Changjiang River water and Li-rich seawater are the only two major contributors of dissolved Li in the mixing zone. This salinity–Li relationship is consistent with observations made in the St. Lawrence estuary and the Gulf of Papua (Stoffyn-Egli, 1982; Brunskill *et al.*, 2003). However, the use of Li concentrations alone does not permit identification of all the processes potentially occurring in the mixing zone. Indeed, as indicated by experimental investigations and by previous studies of Icelandic estuaries, isotopic exchanges may modify riverine dissolved isotopic compositions without significantly affecting elemental concentrations (Jones *et al.*, 2012, 2014). When we report the dissolved $\delta^7\text{Li}$ as a function of the inverse of the Li concentration (Fig. 1b), all the samples are distributed along the theoretical mixing line between the Changjiang River water and seawater. These results *a priori* support the conservative behaviour of the dissolved Li and its isotopes along the studied transect.

The relatively high seawater Li concentration may mask some of the Li release during SPM dissolution (Morin *et al.*, 2015) or Li uptake by SPM (Pogge von Strandmann *et al.*, 2008). The dissolution rates of quartz, albite, and clays, which are major constituent minerals of Changjiang SPM, are relatively slow, because they have undergone intermediate to strong chemical weathering in the catchment. For instance, less than 0.1 % of kaolinite and illite would be expected to dissolve in seawater annually (Jeandel and Oelkers, 2015). As a first approximation, we can assume that 0.1 % of the SPM Li could be released by dissolution in the Changjiang Estuary, although the average residence time of the Changjiang diluted water plume is only 5.4 d (Gu *et al.*, 2012). A mass balance model suggests that, in that case, the dissolved $\delta^7\text{Li}$ would decrease by ~ 0.2 ‰ (see Supplementary Information), which is within

analytical uncertainties. For the possible influences of Li uptake, modelling results suggest that the dissolved $\delta^7\text{Li}$ values would be significantly altered in the maximum turbidity zone where suspended sediment concentration can reach ~ 2 g/L (for detailed calculations, see Supplementary Information and Fig. S-2). This is apparently inconsistent with the conservative behaviour of dissolved Li and $\delta^7\text{Li}$ observed in this study (Fig. 1). Thus, we suggest that the influence of sediment–water interactions on dissolved Li is insignificant in the Changjiang Estuary. Additionally, submarine groundwater discharge (SGD, $0.2\text{--}1.0 \times 10^9 \text{ m}^3 \text{ d}^{-1}$) was estimated to be 6–30 % of the river discharge during the flooding season in the Changjiang Estuary (Gu *et al.*, 2012). Although no Li data were reported for this discharge, our results imply that the SGD plays a small role in the isotope compositions of the Li flux to the East China Sea. A similar conclusion has been drawn by Bagard *et al.* (2015) for assessing the modern Li isotopic budget of the ocean, based on investigation of the Li flux and $\delta^7\text{Li}$ of groundwater in the Ganges–Brahmaputra downstream basin.

The conservative mixing of dissolved Li and Li isotopes observed in the Changjiang Estuary exhibits a nonlinear $\delta^7\text{Li}$ variation as a function of salinity. Indeed, the dissolved $\delta^7\text{Li}$ values increase significantly in the head of the estuary, during the initial mixing between river water and seawater (Fig. 1b). This is because seawater is enriched in Li and isotopically heavy compared to river water. We calculate that the addition of 1 % seawater to the Changjiang River water causes the $\delta^7\text{Li}$ value to increase by ~ 3 ‰ (Supplementary Information). When salinity exceeds 7 ‰, the dissolved $\delta^7\text{Li}$ value remains more or less constant (between ~ 30 ‰ and 31.6 ‰). Due to differences in catchment lithologies, climate regimes, and other environmental parameters, the dissolved Li concentrations and $\delta^7\text{Li}$ values of river waters worldwide yield significant spatial and seasonal variations. Nevertheless, as discussed above, physical mixing of different water masses cannot modify the conservative behaviours of dissolved Li and Li isotopes in estuaries. Therefore, the fast response and significant variation of riverine $\delta^7\text{Li}$ to a small volume of seawater addition observed in this study

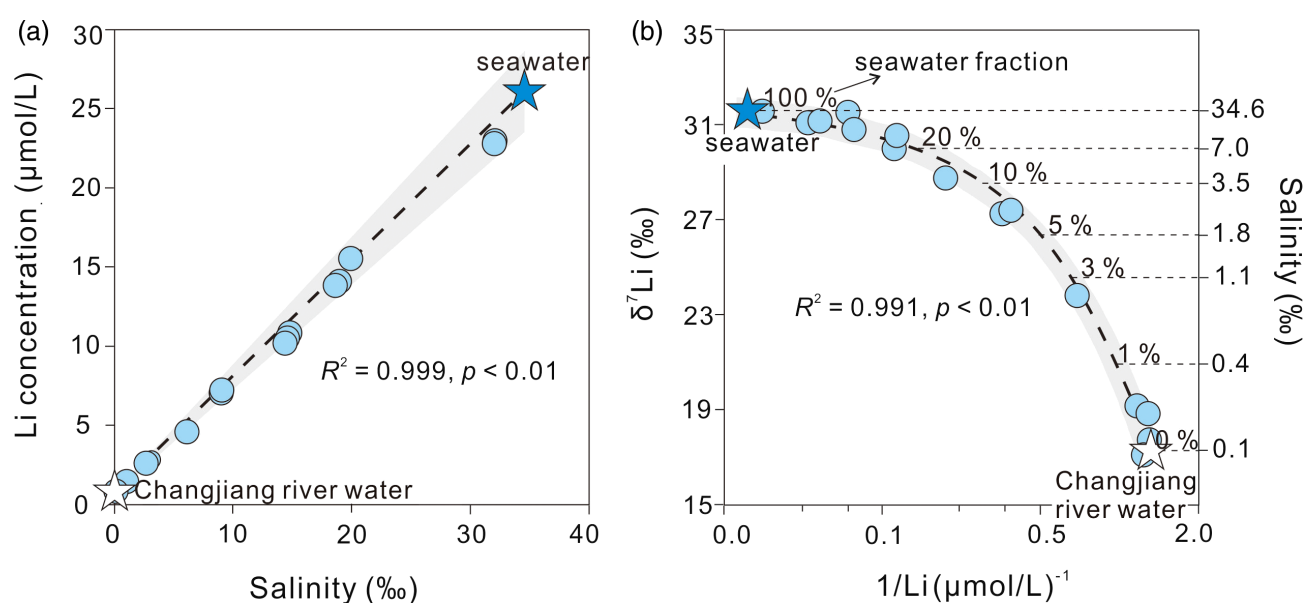


Figure 1 (a) Variations of dissolved Li concentration as a function of salinity, and (b) variations of dissolved $\delta^7\text{Li}$ as a function of the inverse of the molar Li concentration ($1/\text{Li}$). The x-axis in (b) is logarithmic. The dashed lines and shaded areas represent calculated conservative mixing between seawater (dark blue star) and Changjiang River water (white star). The mass balance model is provided in the Supplementary Information. The shaded areas are estimated using the Monte Carlo method, given that analytical uncertainties in Li concentrations and $\delta^7\text{Li}$ are 10 % and 0.6 ‰, respectively. The analytical uncertainty for each $\delta^7\text{Li}$ value is smaller than the symbol size.

is expected to be universal. Indeed, in most large rivers, dissolved Li concentrations are two orders of magnitude lower than that of seawater, and $\delta^7\text{Li}$ values are 5–10 ‰ less (Huh *et al.*, 1998). Caution must therefore be paid to this effect when sampling rivers at their mouths for quantifying their contribution of Li and $\delta^7\text{Li}$ flux to the ocean.

Particulate Li and $\delta^7\text{Li}$ Behaviour in the Estuary

At the XLJ gauging station, SPM Li concentrations, $\delta^7\text{Li}$ values, and suspended sediment concentrations (SSC) exhibit seasonal and depth variations (see Fig. S-4). In contrast, depth profiles performed along the estuarine transect exhibit negligible or small variations, and a noticeable increase can be observed as one moves offshore (Fig. S-5). Generally, depending on the grain size, density, and shape of detrital minerals, hydrodynamic sorting in riverbeds and floodplains may cause mineralogical and geochemical fractionation during SPM transport (Guo *et al.*, 2018). The sediment Al/Si ratio allows us to constrain the effects of hydrological sorting on isotope proxies (Dellinger *et al.*, 2014). As shown in Figure 2a, Li/Si ratios positively correlate with Al/Si ratios for both riverine SPM (orange symbols) and estuarine SPM (blue symbols), suggesting a dominant control on Li concentrations by Al-rich materials (*e.g.*, clays) present in the SPM (Vigier *et al.*, 2008; Dellinger *et al.*, 2014). Nevertheless, the river and estuary data follow significantly different slopes (Fig. 2a). This cannot be attributed to adsorption in the estuary as exchangeable Li accounts for less than 1 % of the SPM Li and thus has a negligible influence on particulate Li (see Supplementary Information). As indicated by previous studies, this difference in slope can be caused either by ongoing weathering (Lupker *et al.*, 2012) or by a change of sediment source (Yang *et al.*, 2019). In the former case, the steeper slope defined by the estuarine SPM data may indicate clay formation in the estuarine environment. However, the formation of clay minerals along the sampling transect is not supported by XRD results

(Table S-3). On the other hand, $\delta^7\text{Li}$ values of SPM samples show no shift toward seawater (Fig. S-3a), in contrast to the altered $^{87}\text{Sr}/^{86}\text{Sr}$ ratios observed in Icelandic estuaries, for instance (Jones *et al.*, 2014). This discrepancy may be explained by the low chemical reactivity of Changjiang-derived SPM, which mainly come from highly weathered and recycled continental sediments and shales. All these observations suggest that SPM Li is not controlled by a chemical process (*e.g.*, water-sediment interactions) in the Changjiang Estuary, and a physical process could be the dominant factor, as discussed below.

More information can be inferred from the $\delta^7\text{Li}$ vs. Li/Al diagram (Dellinger *et al.*, 2017). As shown in Figure 2b, all the river SPM samples are isotopically fractionated compared to their likely parent lithologies because they contain weathering products formed within the basin (Yang *et al.*, 2021). SPM collected in the lower reaches logically represents an average composition of fine particulates from the whole basin. Accordingly, $\delta^7\text{Li}$ and Li/Al variations of SPM collected at the XLJ station are best explained by a simple binary mixing between sediments collected in the upper Changjiang basin and those from the middle basin (Fig. 2b). In contrast, $\delta^7\text{Li}$ and Li/Al ratios of SPM collected along the estuarine transect show a distinct trend. From the C1 site to offshore sites, these values progressively increase towards the binary mixing trend defined between un-weathered shale and igneous rocks (Dellinger *et al.*, 2014). Quartz, feldspar, illite, and kaolinite are four major minerals (*i.e.* each accounting for >10 %) of terrigenous sediments from the Changjiang River (Yang *et al.*, 2002). According to the XRD results, quartz contents in the estuarine SPM increase from ~26 % to ~41 % in the offshore direction, while illite and kaolinite contents both decrease by ~10 % (Table S-3). Thus, the trend observed for estuarine SPM in Figure 2b can be explained by a physical loss of clay minerals and/or a gain of Si-rich primary minerals.

Flocculation and resuspension of fine sediments are two fundamental processes occurring in river estuaries. When encountering alkaline seawater, river-borne clay minerals, especially kaolinite, can be easily aggregated and deposited rapidly

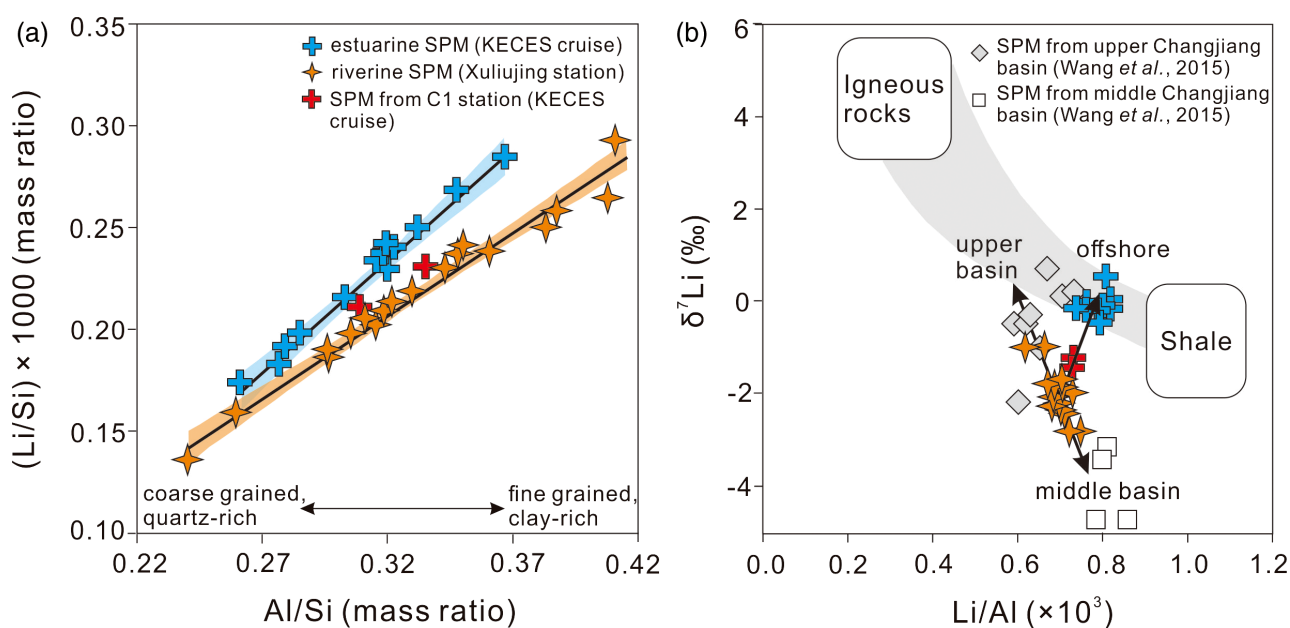


Figure 2 (a) Variations of Li/Si ratios as a function of Al/Si ratios for riverine (orange, including the most inland site C1 in red) and estuarine SPM (blue), and (b) $\delta^7\text{Li}$ versus Li/Al ratios for the same samples. The linear correlations between Li/Si and Al/Si are displayed with 95 % confidence intervals (shaded areas). The igneous rocks and shale endmembers in (b) are modified after Dellinger *et al.* (2014), and the shaded area reflects their mixing. Changjiang River SPM data are from Wang *et al.* (2015).

onto the seafloor. Milliman *et al.* (1985) once estimated that nearly 40 % of the sediment load can be trapped in the Changjiang Estuary during flood season. Seafloor sediments can be resuspended by strong tidal and wind-driven currents, as supported by the several orders of magnitude higher SPM concentration observed at sites C6–C8 than at landward or seaward sites (see Fig. S-5c). Therefore, the offshore transport of SPM in the Changjiang Estuary may result in the preferential flocculation and deposition of clay minerals during the flooding season, while primary minerals or other fine-grained particles tend to be resuspended and carried seaward by currents. Consequently, these physical processes result in a visible increase of SPM $\delta^7\text{Li}$ values (by $\sim 1.2\text{‰}$) from the most inland site (C1) to the most offshore sites.

Implications for Li Isotopes as Tracers of Continental Weathering

Previous studies suggest that both the dissolved and particulate $\delta^7\text{Li}$ values related to clay formation are powerful tracers of weathering processes. Our results demonstrate that their behaviours are visibly decoupled in the estuary. During estuarine flocculation and resuspension of river-borne fine particles, the SPM $\delta^7\text{Li}$ values progressively approach the upper continental crust value. This process is distinct from the general control of hydrodynamic sorting on elemental geochemical compositions during source-to-sink river sediment transport (Lupker *et al.*, 2012; Dellinger *et al.*, 2014). Although the effects of ongoing weathering are negligible in Changjiang-like estuaries, an increase of particulate $\delta^7\text{Li}$ values would be expected in rivers draining basaltic terrains (Pogge von Strandmann *et al.*, 2008). Consequently, the utility of detrital $\delta^7\text{Li}$ values for tracing past continental weathering in coastal and marginal seas seems to be more complicated than for the isotopic values of the dissolved phase (Yang *et al.*, 2021). Caution should therefore be exercised when using $\delta^7\text{Li}$ values, and other similar sediment geochemical proxies, measured in bulk sediments from marginal seas to reconstruct past continental weathering.

Our data along a salinity transect provide the first and clear evidence of the conservative behaviour of dissolved Li and Li isotopes during estuarine mixing in a large, particle-rich river. Differing from the case study on Li isotopes in a small Icelandic estuary (Borgarfjörður) draining dominantly basaltic terrains (Pogge von Strandmann *et al.*, 2008), the large Changjiang basin contains various rock types, including intensely weathered shales and Ca-Mg-depleted sediments. This strongly supports the notion that information on continental weathering carried by the dissolved loads (*i.e.* $\delta^7\text{Li}$) of large rivers can be propagated to the ocean without significant modification. Scavenging processes thus have negligible influence on aqueous Li behaviour, at least in Changjiang-like basins and estuaries, which verifies the assumption of conservative behaviour when using Li isotopes in marine authigenic archives to reconstruct past continental weathering variations and related carbon cycles (Misra and Froelich, 2012). Additionally, it is noteworthy that $\delta^7\text{Li}$ values of estuarine waters significantly increase with seawater addition at low salinities. This highlights the need for precise salinity assessments when sampling estuarine waters for quantifying global Li and $\delta^7\text{Li}$ budgets and continental fluxes to the ocean.

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Additional Information

Supplementary Information accompanies this letter at <https://www.geochemicalperspectivesletters.org/article2133>.



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