

## Enrichment and Release of Rare Earth Elements during Weathering of Sedimentary Rocks in Wujiang Catchments, Southwest China

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Received 14 December 2005; revised 30 January 2006

**Abstract:** Thirteen weathering profiles of sedimentary rocks such as limestone, dolomitic limestone, dolomite, sillicite, black shale and purple sandrock from Wujiang catchments were selected for study on enrichment and release behavior of rare earth elements (REE) during weathering, and its impact on plant growth and riverine REE distribution in the catchments with methods of hierarchical cluster analysis and mass balance calculation in order to set a basis for riverine material source research and agricultural production. The results show that the enrichment degree of REE in calcareous soils from the Wujiang catchments is much higher than that of limestone, yellow soil, upper continental crust (UCC), China soil (CS) and world soil (WS). The ability of enrichment and release of REE is partly controlled by distribution of REE in bedrocks, contents and adsorption ability of organic matters, clay minerals and Fe-oxides/hydroxides in weathering profiles. The REE released from weathering of carbonate rocks and clastic rocks can be absorbed and utilized by local plants. The results also reveal that release of REE and Fe mainly from weathering of carbonate rocks and partly from clastic rocks exerts an important control on riverine REE distribution.

**Key words:** rare earth elements; enrichment; release; weathering; sedimentary rocks

**CLC number:** O614.33; X142      **Document code:** A      **Article ID:** 1002 – 0721(2006)04 – 0491 – 06

Recently, the environmental pollution of rare earth elements (REE) has increased dramatically as a result of wide application of REE in agricultural production as microelement fertilizers as well as in industry<sup>[1-4]</sup>. Therefore, the biogeochemical cycling of REE and its ecological and environmental effects are greatly concerned by many researchers such as Zhu et al.<sup>[3]</sup> Zhang et al.<sup>[4]</sup> and Franca et al.<sup>[5]</sup>. Rock weathering is an important REE source for an ecological system and a key process of global biogeochemical cycling of REE. Thus, researches on geochemical behavior of REE during rock weathering can increase our under-

standing on biogeochemical cycling of REE and its ecological and environmental effects. Compared with other rocks such as granites and basalts, research on geochemical behavior of REE during weathering of sedimentary rocks are not enough. At present, many issues such as enrichment mechanisms, bioavailability, release and its impact on riverine distribution of REE during weathering of sedimentary rocks such as limestone remain unsolved. Weathering profiles of sedimentary rocks were used discuss the above issues in order to improve agricultural production and research on riverine REE sources.

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**Foundation item:** Key Direction Projects of the Innovation Program of Chinese Academy of Sciences (KZCX3-SW-140, KZCX2-105)

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## 1 Materials and Methods

The Wujiang River drains from the Yunnan-Guizhou Plateau, and is the largest tributary of the Yangtze River. The major lithologies are limestone in the upper reaches, dolomite and clastic rocks in the rest areas (Fig. 1). The region has a complex topography ranging in elevation from 300 to 2300 m, and a sub-tropical and humid monsoonal climate with a mean annual average temperature of 9 ~ 17 °C and mean annual precipitation of 900 ~ 1400 mm. The upper reaches are mainly covered with calcareous soils and shrubs, while the rest areas are covered with calcareous and yellow soils and arbor-shrub vegetation.

In order to investigate weathering of limestone and other sedimentary rocks, we sampled weathering profiles of limestone (8), dolomitic limestone (1), dolomite (1), silicalite (1), black shale (1) and purple sandrock (1) along Wujiang River (Fig. 1 and Table 1). Profile locations were selected in slight concavities within gently convex upland positions to minimize physical erosion and local mixing<sup>[9]</sup>. All weather-

ing profiles were dug by hand to hard rock. Profile samples were horizontally collected from bottom to top.

Air-dried samples were ground in agate mortar into 20 meshes for pH analysis and 200 meshes for other measurements. The pH values of soil samples were determined by a pH meter with a soil/water mass ratio of 1/2.5. The 200 mesh samples were dried at 120 °C for 3 h and used for bulk chemical analysis and trace element determination. Bulk chemical analysis was completed with traditional wet chemical methods while trace element contents were determined by inductively coupled plasma mass spectrometry (ICP-MS; ELEMENT, Finnigan MAT, UK), using Rh as an internal standard after acid (HNO<sub>3</sub> + HF) digestion in PTFE bombs<sup>[10]</sup>. Monitored with in-house standards of quartz sandstone (GBW07106), limestone (GBW07120), calcareous soil (GBW07404) and yellow soil (GBW07405), the precision is better than 2% for major elements and 8% for trace elements, respectively. As average shale (PAAS)<sup>[11]</sup> represents average composition of upper continental crust and was frequently used to normalize the distribution of REE, da-

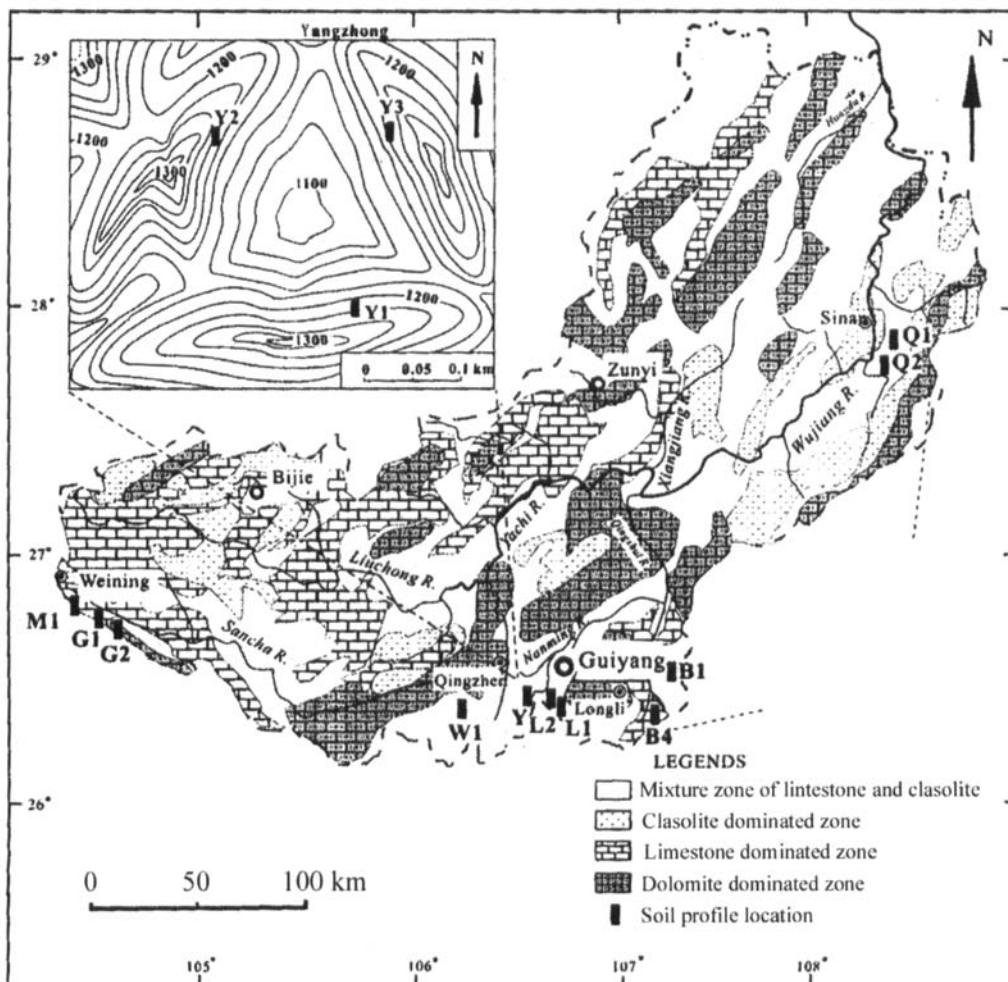


Fig. 1 Geological map showing locations of weathering profiles

ta of samples and PAAS were used to calculate Enrichment Factor (EF) of elements in samples ( $EFX = X_{\text{sample}}/X_{\text{PAAS}}$ , where  $X_{\text{sample}}$  and  $X_{\text{PAAS}}$  are contents of element  $X$  in samples and PAAS). R-type hierarchical cluster analysis was completed with the software of SPSS 11.5 using average linkage (between groups) and Pearson correlation.

## 2 Results

Table 1 shows bulk chemical composition and related parameters of bedrocks and average soil profiles. For weathering profiles of limestone (Q1 ~ M1) and dolomitic limestone (W1), the neutral-acidic soils have much lower content of CaO and higher contents of SiO<sub>2</sub>, TiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub> and loss of ignition (LOI)

than the bedrocks. For dolomite weathering profile (G2), the contents of CaO and MgO decrease dramatically and the contents of other components such as SiO<sub>2</sub>, TiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, Fe<sub>2</sub>O<sub>3</sub> and LOI increase significantly from bedrock to topsoil. For weathering profiles of sillicalite (B1), shale (L1) and sandstone (L2), the bedrocks are mainly composed of SiO<sub>2</sub>, and the acidic soils have a similar composition with their respective bedrocks.

Relative to PAAS, the Enrichment Factor of REE (EFREE) and shale-normalized ratio of La/Yb ((La/Yb)<sub>SN</sub>) of bedrocks and average weathering profiles were calculated from Table 1. The results are shown in Fig.2(a, b). Fig.2(a) shows that EFREE (1.19 ~ 1.85) of calcareous soils (Q1 ~ G2) is significantly

**Table 1 Bulk chemical data and related parameters of bedrock and average soil profiles compared with China soil (CS), world soil (WS) and upper continental crust (UCC)\***

Bedrock types	Profile No.	Depth/ m	Sample type	pH	La/Yb	REE/10 <sup>-6</sup> (%)														
						g* g <sup>-1</sup>	LOI <sup>g</sup>	SiO <sub>2</sub>	TiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MnO	MgO	CaO	Na <sub>2</sub> O	K <sub>2</sub> O	P <sub>2</sub> O <sub>5</sub>			
Limestone	Q1	0.82	R <sup>e</sup>		32.108	36.938	0.15	2.97	0.67	0.16	0.44	0.04	0.42	52.74	0.07	0.09	0.23			
			AS(3) <sup>f</sup>	6.72	15.045	280.916	13.50	54.90	2.03	16.65	8.43	0.17	1.03	1.35	0.20	1.04	0.35			
	Q2	0.62	R		14.332	10.579	0.15	4.31	0.53	0.16	0.23	0.01	0.40	52.54	0.10	0.01	0.30			
			AS(3)	6.69	8.160	326.199	13.79	51.15	2.08	17.59	10.16	0.18	1.22	1.59	0.15	1.41	0.35			
	B4	1.02	R		5.258	7.891	0.10	3.57	0.53	0.16	0.34	0.05	0.30	52.69	0.17	0.06	0.32			
			AS(4)	7.06	15.839	232.670	15.03	61.98	2.33	10.30	5.38	0.24	0.81	1.99	0.13	0.96	0.37			
	Y1	0.71	R		9.463	5.195	0.10	3.20	0.47	0.15	0.16	0.01	0.84	52.10	0.07	0.06	0.23			
			AS(3)	6.50	19.141	341.985	12.79	66.33	1.55	12.06	4.19	0.14	0.60	1.09	0.11	0.33	0.36			
	Y2	0.31	R		6.933	2.292	0.21	3.15	0.47	0.16	0.28	0.01	0.24	52.51	0.04	0.01	0.23			
			AS(2)	5.53	15.380	237.029	25.58	46.77	1.15	16.10	5.33	0.15	0.90	2.33	0.07	0.42	0.50			
Y3	0.62	R		12.592	1.399	0.45	1.73	0.73	0.15	0.13	0.01	0.38	53.56	0.04	0.04	0.27				
		AS(2)	6.18	15.297	308.188	14.18	46.74	1.52	21.19	6.34	0.11	1.44	4.52	0.10	0.38	0.45				
G1	0.52	R		4.440	2.594	0.15	2.70	0.47	0.16	0.16	0.05	0.37	53.60	0.07	0.05	0.30				
		AS(2)	6.54	12.963	337.406	16.85	52.31	1.55	19.43	6.08	0.11	0.90	1.56	0.14	0.33	0.40				
M1	1.57	R		6.330	7.204	0.20	3.01	0.77	2.12	0.47	0.04	0.83	51.09	0.07	0.68	0.26				
		AS(3)	6.67	12.632	220.710	13.56	59.24	1.27	14.85	6.21	0.20	1.37	1.14	0.13	0.99	0.40				
Dolomitic limestone	W1	0.37	R		6.499	2.321	0.10	2.10	0.50	0.16	0.16	0.01	4.31	49.41	0.07	0.05	0.23			
			AS(2)	6.01	15.230	319.938	20.09	49.26	1.66	18.46	6.28	0.21	1.01	1.20	0.13	0.72	0.44			
Dolomite	G2	0.72	R		4.917	4.702	0.20	2.09	0.87	0.16	0.17	0.01	20.84	32.18	0.11	0.01	0.21			
			AS(3)	6.73	11.201	277.336	14.50	49.96	1.67	20.69	6.66	0.24	2.254	2.39	0.191	0.717	0.41			
Sillicalite	B1	1.40	R		13.748	107.001	3.90	83.12	1.30	4.94	4.77	0.05	0.32	0.28	0.09	0.44	0.70			
			AS(8)	4.69	15.157	115.114	6.57	74.50	1.70	8.59	6.69	0.03	0.421	0.38	0.126	0.537	0.31			
Black shale	L1	0.72	R		8.329	141.381	7.91	59.96	1.47	19.22	7.02	0.03	1.22	0.19	0.25	2	0.41			
			AS(3)	4.57	10.370	156.322	9.70	63.66	1.04	18.18	4.78	0.03	0.72	0.08	0.149	1.292	0.32			
Purple sandrock	L2	0.82	R		11.916	88.142	1.59	89.40	0.80	5.77	1.46	0.04	0.09	0.15	0.01	0.26	0.25			
			AS(3)	4.78	7.223	81.962	10.00	59.31	1.20	21.96	5.66	0.06	0.258	0.24	0.265	0.34	0.34			
China soil <sup>a</sup>	CS				15.660	155.190			0.63	12.56	4.25	0.07	1.23	1.30	1.50	2.26	0.00			
World soil <sup>b</sup>	WS				13.333	153.800		71	0.8	13.4	6	0.1	0.8	2.1	0.7	1.7	0.2			
Crust <sup>c</sup>	UCC				13.636	146.370		66.0	0.5	15.2	5.0	0.1	2.2	4.2	3.9	3.4	0.2			
Shale <sup>d</sup>	PAAS				13.546	184.773														

\* a, b: After Chen<sup>[12]</sup>; c: After Taylor and McLennan<sup>[13]</sup>; d: After McLennan et al.<sup>[11]</sup>; e: Bedrock; f: Depth-weighted average, the number in brackets represents sample number used to average; g: Loss of ignition, a measure of contents of organic matters

higher than 1, and higher than that of carbonate bedrocks (Q1 ~ G2), China soil (CS)<sup>[12]</sup>, world soil (WS)<sup>[12]</sup> as well as upper continental crust (UCC)<sup>[13]</sup>, indicating that REE is significantly enriched during carbonate weathering. Fig. 2 (a) also shows that EFREE of yellow soils from weathering profiles of sillicite (B1) and shale (L1) is slightly higher than that of their respective bedrocks, but still lower than 1 and that of UCC. Sandrock (L2) has an EFREE lower than sillicite (B1), shale (L1) and 1, but higher than its purple soils. Fig. 2(a, b) reveals that limestone from Q1 and Q2 has a  $(La/Yb)_{SN}$  (1.05 ~ 2.37) higher than 1, UCC and calcareous soils implying that HREE is preferentially enriched during weathering. Bedrocks from weathering profiles of limestone (B4 ~ M1), dolomitic limestone (W1) and dolomite (G1) have a low  $(La/Yb)_{SN}$  (0.32 ~ 0.93), but the  $(La/Yb)_{SN}$  increases significantly from bedrock to topsoil implying that LREE is preferentially enriched during weathering. Weathering profiles of sillicite (B1) and shale (L1) have similar  $(La/Yb)_{SN}$  variation trends with most carbonate weathering profiles (B4 ~ G2). The variation trend of  $(La/Yb)_{SN}$  in weathering profile of sandrock (L2) is similar to those of Q1 and Q2.

As REE can be divided into LREE and HREE, it is necessary to show behavior of REE together with LREE and HREE when the behavior of REE is studied. R-type hierarchical cluster analysis for bedrocks and profiles shown in Fig. 3(a, b) shows that there are good correlations among LREE, REE, HREE, LOI, Fe, Ti, Al and K in bedrocks and moderate cor-

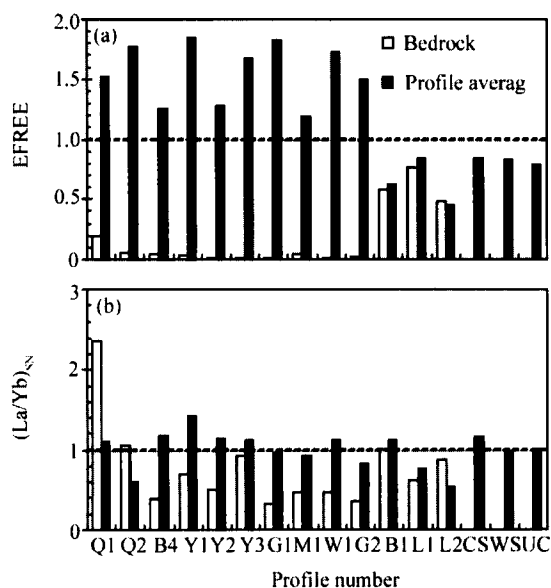


Fig. 2 PAAS normalized enrichment factor of REE (EFREE) (a) and PAAS-normalized La/Yb (b) for bedrocks and depth-weighted average of profiles ( $(La/Yb)_{SN} = (EF-La)/(EF-Yb)$ )

relations among LREE, REE, HREE, Fe, Ti and Al in profiles.

### 3 Discussion

#### 3.1 Enrichment mechanisms of REE

LOI is a measure of contents of organic matters in samples. The good correlations among LREE, REE, HREE, LOI, Fe, Ti, Al and K in bedrocks (Fig. 3 (a)) indicate that LREE and HREE probably are hosted in common phases such as adsorbed by organic matters, Fe-oxides/hydroxides, clay minerals and/or fixed within crystal lattice of ilmenite. As Ti is almost immobile during rock weathering<sup>[7]</sup>, the moderate correlations among LREE, REE, HREE, Fe, Ti and Al in weathering profiles (Fig. 3(b)) imply that a small proportion of the released REE is leached away while a major part of it is fixed by clay minerals and Fe-oxides/hydroxides.

In order to learn enrichment mechanisms of REE in weathering profiles, we calculated Mobility Factor ( $MF$ ) of elements<sup>[7]</sup> ( $MF_X = (X/TiO_2)_{sample} / (X/TiO_2)_{bedrock}$ , where  $(X/TiO_2)_{sample}$  and  $(X/TiO_2)_{bedrock}$  represent mass ratio of  $X/TiO_2$  in profile samples and bedrock samples, respectively.). The results are shown in Fig. 4. Fig. 4 together with enrichment char-

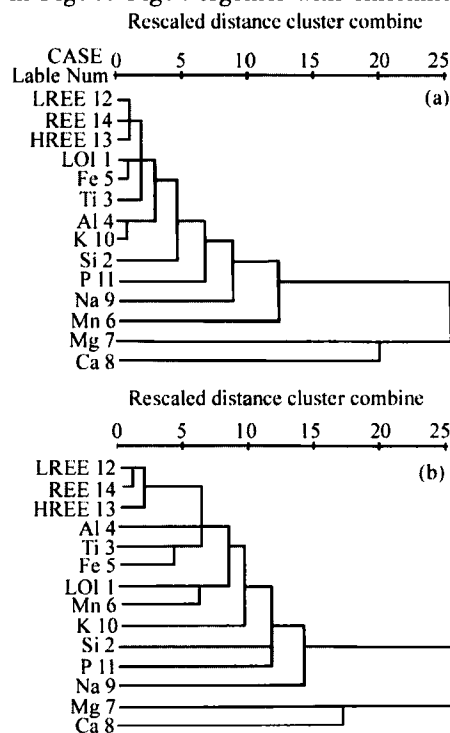


Fig. 3 Dendrograms showing results of R-type hierarchical cluster analysis for LREE, REE, HREE and other components (a) Bedrock samples; (b) Weathering profile samples

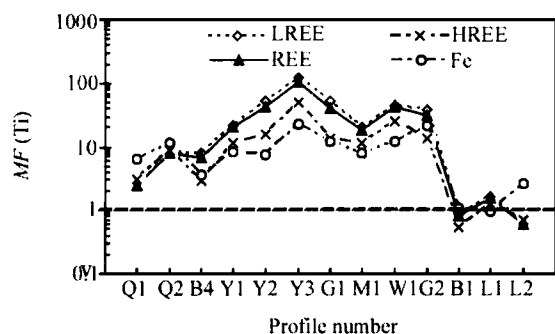


Fig.4 Depth-weighted average of Mobility Factor ( $MF$ ) for LREE, REE, HREE and Fe in profile samples relative to their respective bedrock samples assuming the immobility of Ti

acteristics of REE in weathering profiles shown in Figs. 2 and 3 reveals that the  $MF$  values of LREE, REE, HREE and Fe in all carbonate weathering profiles share the same variation trend and are higher than 1, indicating that the enrichment of LREE, REE and HREE results from a supergene enrichment mechanism caused by continual cycling of REE from leaching zone to a deeper zone of accumulation and subsequent erosion of the leached zone. The  $MF$  values of LREE, REE and HREE in shale weathering profile (L1) are slightly higher than 1, implying that the weak enrichment of LREE, REE and HREE is a result of weak supergene enrichment of REE. The  $MF$  values of LREE, REE and HREE in weathering profiles of sillicite (B1) and sandrock (L2) are lower than 1 implying that the weak enrichment of REE in profile B1 is a result of weak residual enrichment of REE caused by preferential leach of other more soluble components such as Si, while the loss of REE in profile L2 is caused by leach of REE away from the profile. It can be inferred from Figs. 2, 3 and 4 that the fractionation characteristics of REE (Fig. 2(b)) during carbonate weathering are mainly controlled by (La/Yb)<sub>SN</sub> in bedrocks, adsorption capacity of organic matters, Fe-oxides/hydroxides and clay minerals, that preferential enrichment of LREE during weathering of sillicite (B1) and shale (L1) may have something to do with its stronger fixation by Fe-oxides/hydroxides than HREE due to larger ion radius of the former, and that the preferential loss of LREE in sandrock weathering profile is probably caused by leach of REE (especially LREE) in the form of silicic complex.

### 3.2 Release of REE and plant growth

Calcareous soils are frequently characterized by a high base status and pH between 7.5 and 8.5, and by low bioavailability of essential elements because of low

solubility of these elements under calcareous soil conditions<sup>[14]</sup>. However, the floristic composition of calcareous and acid silicate soils differs markedly. Calcicole plants developed on calcareous soils can adapt to calcareous conditions mainly by: (1) colonizing roots with effective mycorrhizal fungi to promote greater soil volume exploitation and enhanced essential element uptake<sup>[15, 16]</sup>; (2) secreting a large amount of root exudates including root organic acid exudates, mainly di- and tricarboxylic organic acids or anions of these acids to promote mineral dissolution, organic matter mineralization, and root uptake of barely soluble essential element pools from the rhizosphere<sup>[15, 17]</sup>. Our calcareous soils are characterized by a weak acid to neutral pH (5.5 ~ 7.5), low base status, and high enrichment of REE relative to UCC, CS and WS (Table 1, Fig. 2). Furthermore, our cluster analysis (Fig. 3(b)) reveals that REE is mainly adsorbed by various components. Therefore, under natural conditions, calcicole plants in our studied areas can release and utilize the enriched REE within rhizosphere by secreting a large amount of root organic acid exudates, mainly di- and tricarboxylic organic acids or anions of these acids. The REE released from weathering of sillicite, shale and sandrock can also be absorbed and utilized by local plants due to moderate to high enrichment (Fig. 2(a)) and mobility (Fig. 4) of REE during weathering. However, further work is required to examine composition, amount and extraction efficiency of root exudates in order to quantify the amount of REE weathering of sedimentary rocks can supply to local plants.

### 3.3 Impact of weathering on distribution of riverine REE

REE enrichment characteristics and mechanism analysis reveal that the released REE from weathering of carbonate rocks (mainly limestone), sillicite and shale is mainly stored in profiles. The content of REE in sandrock is low, thus only a small amount of REE can be released and leached away during sandrock weathering. Therefore, only a small amount of REE can be released from rock weathering and leached into Wujiang River. Furthermore, the amount of Fe released from weathering of carbonate rocks (mainly limestone) increases from the upper to the lower reaches (G2 ~ Q1) of Wujiang River (Figs. 1 and 4) indicating that the concentration of dissolved and colloidal Fe may increase from the upper to the lower reaches. As Fe-colloid can preferentially adsorb LREE, the concentration of dissolved REE and ratio of dissolved (La/Yb)<sub>SN</sub> decrease from the upper to the

lower reaches. The above analysis can be used to explain the phenomena reported by Han et al.<sup>[19]</sup> that the concentration of dissolved REE of Wujiang River is much lower than that of other low-pH rivers from the rest of the world and that  $(La/Yb)_{SN}$  decreases from the upper to the lower reaches.

## 4 Conclusion

In comparison with CS, WS, UCC and bedrocks, calcareous soils developed from carbonate rocks, yellow soils developed from sillicite and shale, and purple soil developed from purple sandrock show high enrichment, moderate enrichment and slight loss of REE, respectively. Enrichment of REE in carbonate weathering profiles is a result of supergene enrichment of REE during carbonate weathering. Fractionation characteristics of REE during carbonate weathering are mainly controlled by  $(La/Yb)_{SN}$  in bedrocks and fixation ability of REE by organic matters, clay minerals and Fe-oxides/hydroxides in weathering profiles. Residual enrichment of REE (especially LREE) in sillicite weathering profile is controlled by other more soluble elements such as Si and fixation of REE by some components such as Fe-oxides/hydroxides during weathering. The supergene enrichment of REE (especially LREE) in shale weathering profile is controlled by preferential fixation of REE (especially LREE) by components such as Fe-oxides/hydroxides during weathering. The preferential loss of REE (especially LREE) is probably caused by leach of REE (especially LREE) in the form of silicic complex. The REE released from weathering of sedimentary rocks can be absorbed and utilized by calcicole plants. The REE and Fe released from weathering of sedimentary rocks may exert an important control on distribution of riverine dissolved REE.

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