

Supplementary Information (SI) for

~~*In situ* interactive characteristics of highly reactive minerals in soil
colloids and carbon preservation differentially revealed by
nanoseal secondary ion mass spectrometry and X-ray absorption
fine structure spectroscopy~~

New strategies for submicron characterization the carbon binding of reactive
minerals in long-term contrasting fertilized soils: Implications for soil carbon
storage

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15 **Fig. S1** Field fertilization layout and extracted soil (Ferralic Cambisol) colloids from three 24-year (1990-2014) long-term fertilization treatments. Control, no fertilization; NPK, chemical nitrogen, phosphorus and potassium fertilization; NPKM, chemical NPK plus swine manure fertilization.

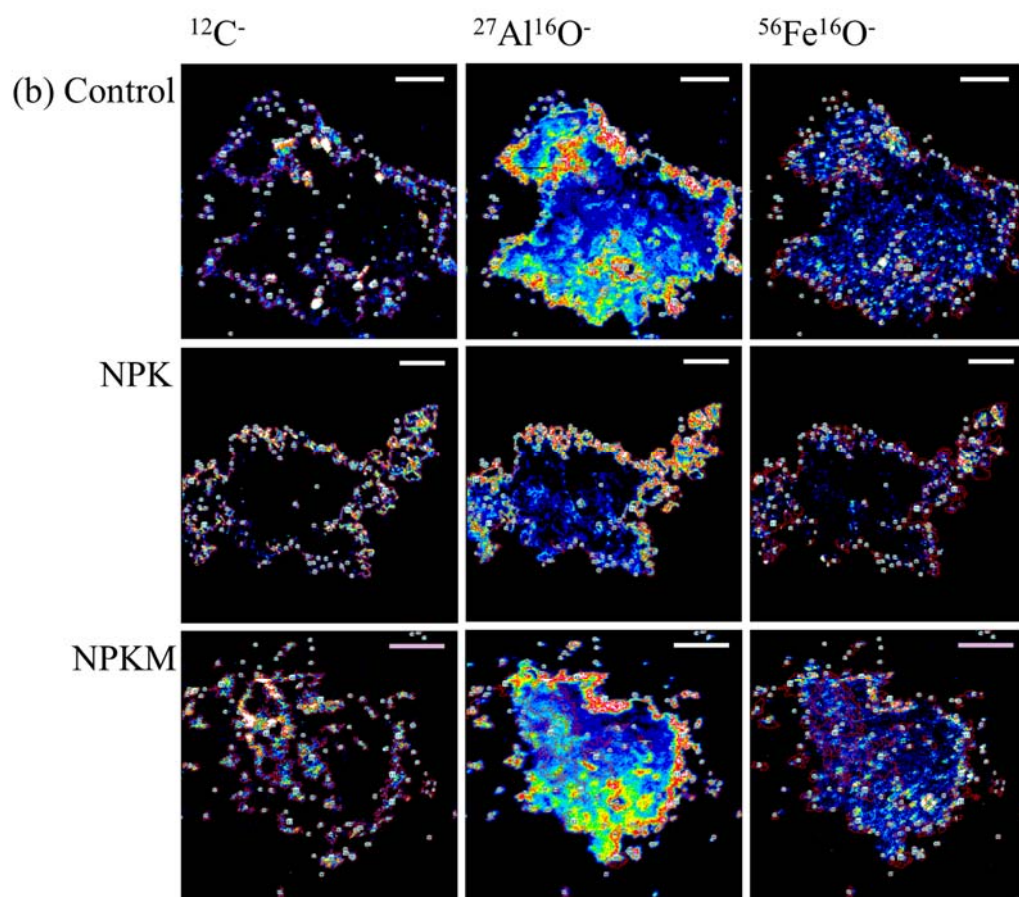
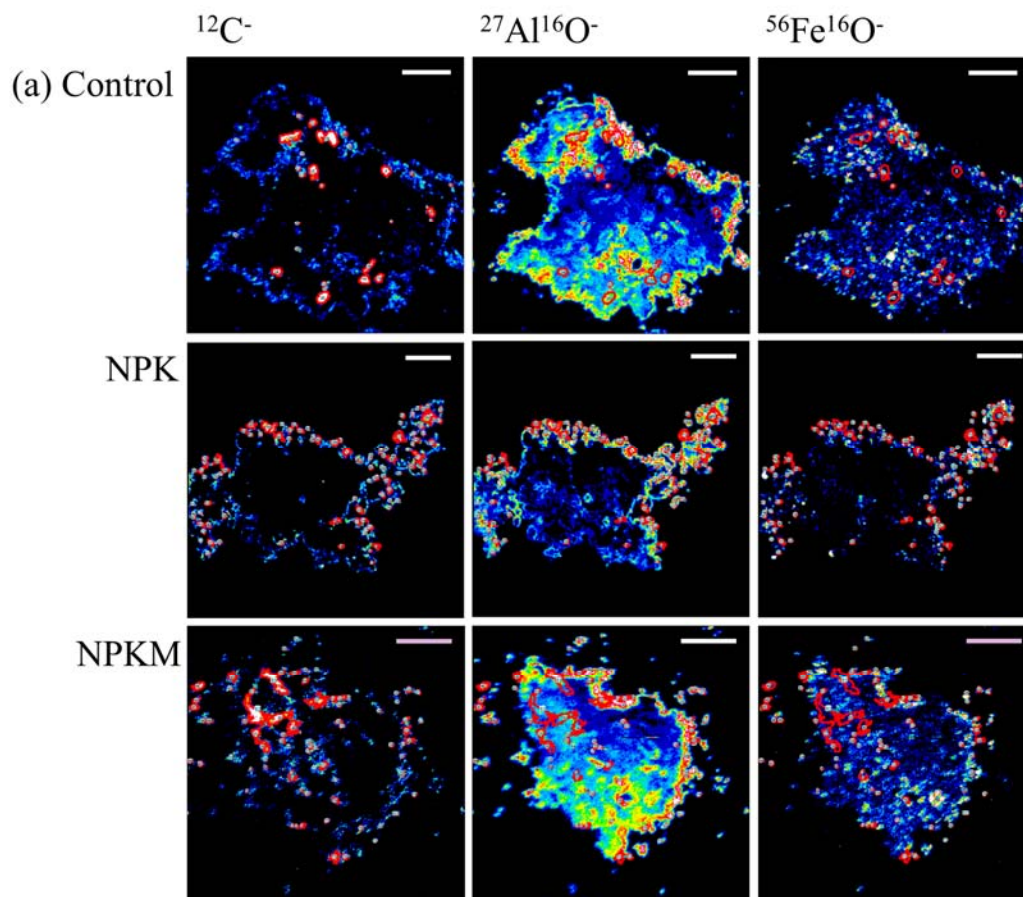


Fig. S2 Region of interests (ROIs) (circled by red line) of $^{12}\text{C}^-$, $^{27}\text{Al}^{16}\text{O}^-$ and $^{56}\text{Fe}^{16}\text{O}^-$ presenting on the NanoSIMS images in soil (Ferralic Cambisol) colloids extracted from three 24-year (1990-2014) long-term fertilization treatments. NanoSIMS images of (a) the $^{12}\text{C}^-$ rich ROIs of $^{12}\text{C}^-$, $^{27}\text{Al}^{16}\text{O}^-$ and $^{56}\text{Fe}^{16}\text{O}^-$ ~~and of~~ (b) the $^{12}\text{C}^-$ less rich ROIs of $^{12}\text{C}^-$, $^{27}\text{Al}^{16}\text{O}^-$ and $^{56}\text{Fe}^{16}\text{O}^-$. Control, no fertilization, $28 \times 28 \mu\text{m}^2$; NPK, chemical nitrogen, phosphorus and potassium fertilization, $30 \times 30 \mu\text{m}^2$; NPKM, chemical NPK plus swine manure fertilization, $25 \times 25 \mu\text{m}^2$. Note that the color intensity calibration bar displayed in the chemical maps corresponds to the relative concentrations of individual elements, but cannot be used to compare one element to another. Bar = $5 \mu\text{m}$.

32 **Table S1. Annual fertilization rates between 1990 and 2014 in Qiyang, China ^a.**

Treatment	Wheat				Corn			
	N (kg ha ⁻¹)	P	K	Swine manure	N (kg ha ⁻¹)	P	K	Swine manure
		(kg ha ⁻¹)	(kg ha ⁻¹)	(Mg ha ⁻¹)		(kg ha ⁻¹)	(kg ha ⁻¹)	(Mg ha ⁻¹)
Control	0	0	0	0	0	0	0	0
NPK	27	16	31	0	63	37	73	0
NPKM	90	16	31	10-15	210	37	73	25-35

^aNote: N fertilizer was as urea, P as calcium superphosphate, K as KCl. Swine manure was calculated in fresh weight. Control, no fertilization; NPK, chemical nitrogen, phosphorus and potassium fertilization; NPKM, chemical NPK plus swine manure fertilization.

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34 **Table S2. Quantification of $^{12}\text{C}^-$ rich ($^{12}\text{C}^-$ -R) and $^{12}\text{C}^-$ less-rich ($^{12}\text{C}^-$ -LR) region of interests (ROIs) ^a**

	Replicates	$^{12}\text{C}^-$ Rich ROIs			$^{12}\text{C}^-$ Less-Rich ROIs		
		ROIs	ROIs Area	Intensity	ROIs	ROIs Area	Intensity
Treatment	Number	Number	/Total area (%)	(Pixel)	Number	/Total area (%)	(Pixel)
Control	8	312	7.47	>90	457	40.18	40-90
NPK	6	567	10.80	>90	532	27.64	40-90
NPKM	6	479	8.23	>50	596	37.99	30-50

^aNote: Control, no fertilization; NPK, chemical nitrogen, phosphorus and potassium fertilization; NPKM, chemical NPK plus swine manure fertilization.

35 **Table S3. Fe mineral standards used in the fitting of Fe K-edge XANES spectra**

Fe mineral standards	Mineral type	Chemical formula	Origin	References
Ferrous sulfate	Inorganic ferrous oxides	$\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$	Aladdin, CAS:7782-63-0	-
Ferrous oxalate	Organic ferrous oxides	$\text{FeC}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$	Aladdin, CAS:6047-25-2	-
Ferric sulfate	Inorganic ferric oxides	$\text{Fe}_2(\text{SO}_4)_3 \cdot 2\text{H}_2\text{O}$	Aladdin, CAS:10028-22-5	-
Ferric oxalate	Organic ferric oxides	$\text{Fe}_2(\text{C}_2\text{O}_4)_3 \cdot 5\text{H}_2\text{O}$	Aladdin, CAS: 2944-66-3	-
Goethite	Iron Oxide	$\alpha\text{-FeOOH}$	Synthetic	Schwertmann & Cornell (2007b)
Hematite	Iron Oxide	$\alpha\text{-Fe}_2\text{O}_3$	Synthetic	Yen et al. (2002) Schwertmann & Cornell (2007c)
Ferrihydrite	Iron Oxide	$\text{Fe}_5\text{HO}_8 \cdot 4\text{H}_2\text{O}$	Synthetic	Michelet al. (2007) Schwertmann & Cornell (2007a)
Maghemite	Iron Oxide	$\gamma\text{-Fe}_2\text{O}_3$	Synthetic	Wang et al. (2008) Schwertmann & Cornell (2007d)

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