Biogeosciences Discuss.,
doi:10.5194/bg-2015-625-AC2, 2016

Interactive comment on "*In situ* interactive characteristics of reactive minerals in soil colloids and soil carbon preservation differentially revealed by nanoscale secondary ion mass spectrometry and X-ray absorption fine structure spectroscopy" *by* Jian Xiao et al.

Jian Xiao et al.

yuguanghui@njau.edu.cn

Received and published: 12 April 2016

Referee #3 Interactive comment on "In situ interactive characteristics of reactive minerals in soil colloids and soil carbon preservation differentially revealed by nanoscale secondary ion mass spectrometry and X-ray absorption fine structure spectroscopy" Jian Xiao et al. yuguanghui@njau.edu.cn Received and published: 15 March 2016

Response to Referee #3 We thank Referee #3 for the exciting and thoughtful com-

ments. We have revised our manuscript in response to the suggestions. All of the revised parts were colored in red in the revised manuscript. Interactive comment on "In situ interactive characteristics of reactive minerals in soil colloids and soil carbon preservation differentially revealed by nanoscale secondary ion mass spectrometry and X-ray absorption ïňAne structure spectroscopy" by Jian Xiao et al.

Anonymous Referee #3 Received and published: 4 April 2016 I recommend the manuscript to be published after minor revisions. This is an excellent research that employs a combination of cutting-edge techniques (NanoSIMS, EXAFS) to explore the mineral-carbon association in soil colloids. The results would be of great signiïňAcance to improve current understanding of the soil C pool and its stability towards global changes. However, I have two comments on the manuscript. First, the effect of long-term fertilization on the Al and Fe mineralogy was not fully discussed, although the nano-SIMS revealed that Fe(Al) and C are coupled. I wonder if chemical extraction experiments could help address the changes of Fe, Al speciation during the long-term fertilization. Second, I would recommend the author put Fig. 5 to the supporting information, as they already have the EXAFS data in Fig. 6 and the Fe EXAFS data are believed to be more informative and quantitative than the XANES data.

Response: Great comments! Our previous studies have shown that long-term fertilization could affect the Al and Fe mineralogy using chemical extraction methods, including the acid ammonium oxalate extraction (Alo and Feo), pyrophosphate extraction(Alp and Fep) and citrate-bicarbon-ate-dithionite (CBD) solution (Fed) (Huang et al., 2016; Wen et al., 2014; Zhang et al., 2013). Specifically, Zhang et al. (2013) observed that the oxalate-extractable Fe (Feo) content of NPKM and M treatments was greater than that of N and NPK treatments in the 20–40 cm layer, but there was no statistical difference between the manure treatments (NPKM and M) and mineral fertilizer treatments (N and NPK) at 0–20 cm. Meanwhile, the pyrophosphate-extractable Fe (Fep) concentrations were less in the NPKM and M treatments than those in the N and NPK treatments at 0–20 cm. Using the citrate-bicarbonate-dithionite (CBD) extraction method, Huang et al. (2016) showed that organic fertilization treatments (NPKM and M) increased the iron freeness index (i.e., the Fed/Fet ratio) when compared to chemical fertilization treatment (NPK). In addition, Wen et al. (2014) found that compared with chemical fertilization (N and NPK), organic fertilization (NPKM and M) significantly ($P < 0.05$) increased amorphous Al and decreased exchangeable Al, while the addition of lime (N with lime and NPK with lime) significantly ($P < 0.05$) increased weakly organically bound Al and decreased exchangeable Al. In the revised manuscript, we added the corresponding discussion and colored in red in the revised manuscript. Based on the suggestion of Referee 3, we had put Fig. 5 to the supporting information (as Fig. S3).

Please also note the supplement to this comment:
http://www.biogeosciences-discuss.net/bg-2015-625/bg-2015-625-AC2-supplement.pdf

Interactive comment on Biogeosciences Discuss., doi:10.5194/bg-2015-625, 2016.

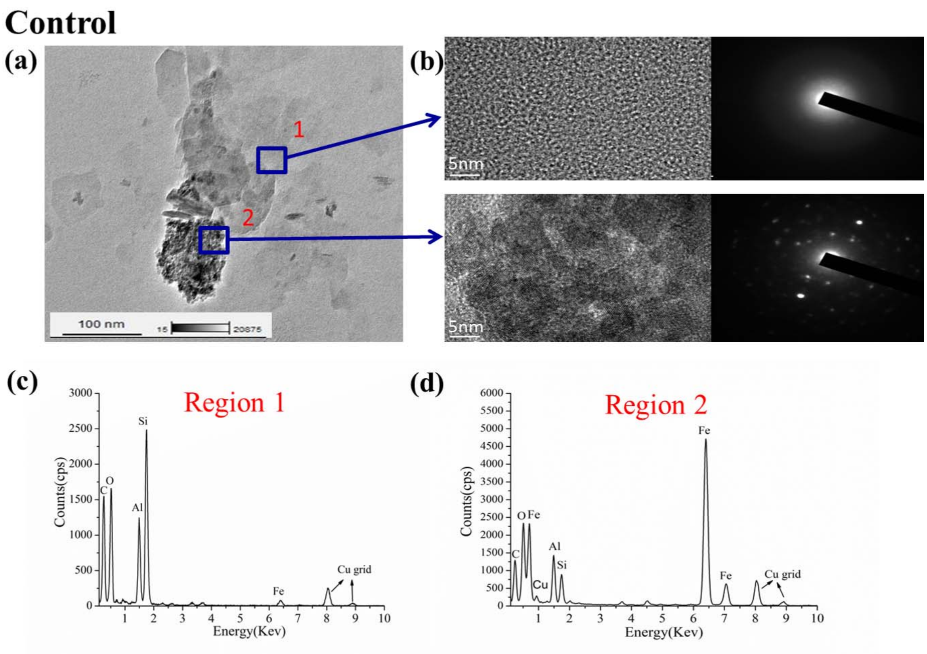

Fig. 1. High-resolution transmission electron microscopy (HRTEM) images of highly reactive minerals from colloids

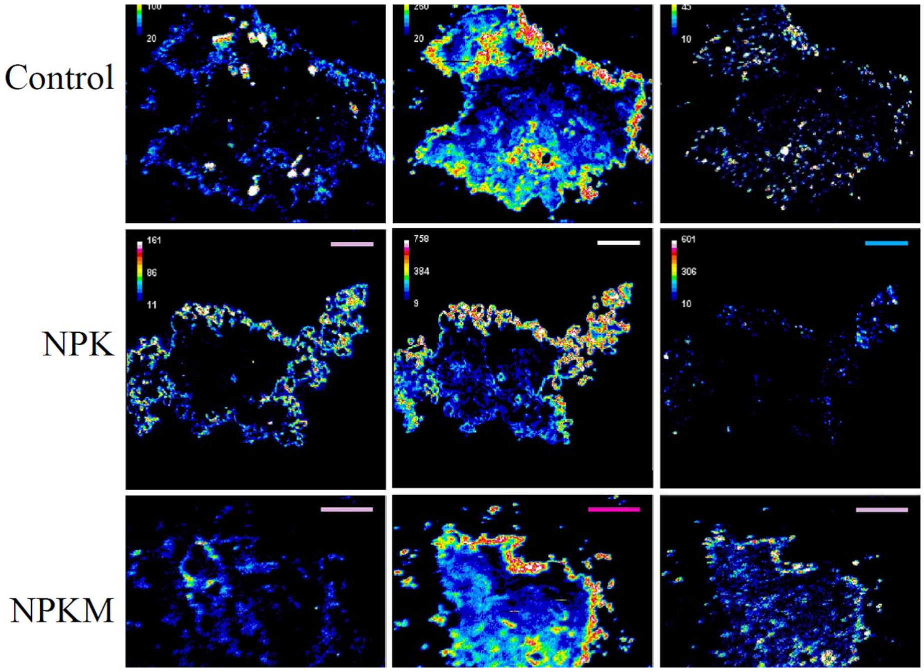

Fig. 2. Representative NanoSIMS images of 12C-, 27Al16O- and 56Fe16O- in soil colloids from three contrasting long-term (1990-2014) fertilization treatments

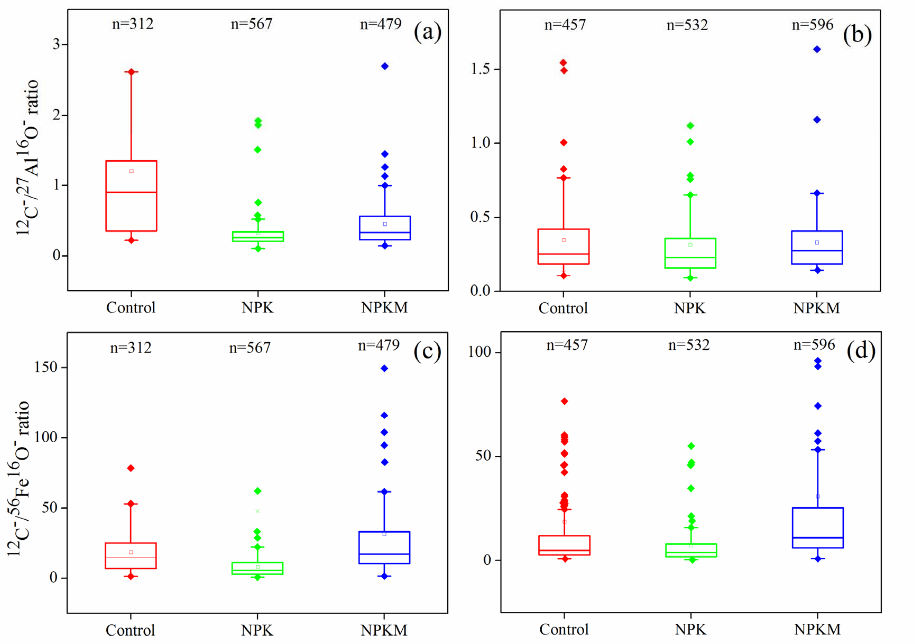

Fig. 3. Box plots of 12C-/27Al16O- (a, b) and 12C-/56Fe16O- (c, d) ratios reflecting the 12C-rich ROIs (a, c) and 12C- less rich ROIs (b, d) of the soil colloids

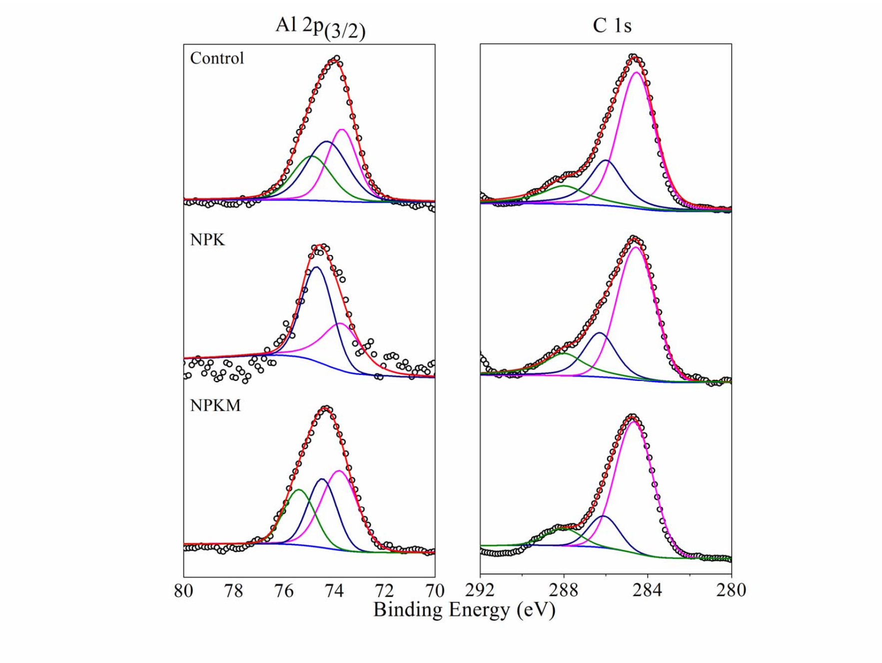

Fig. 4. XPS peak-fitting (Al 2p3/2, C 1s) images recorded from soil (Ferralic Cambisol) colloids extracted under three long-term (1990-2014) fertilization treatments

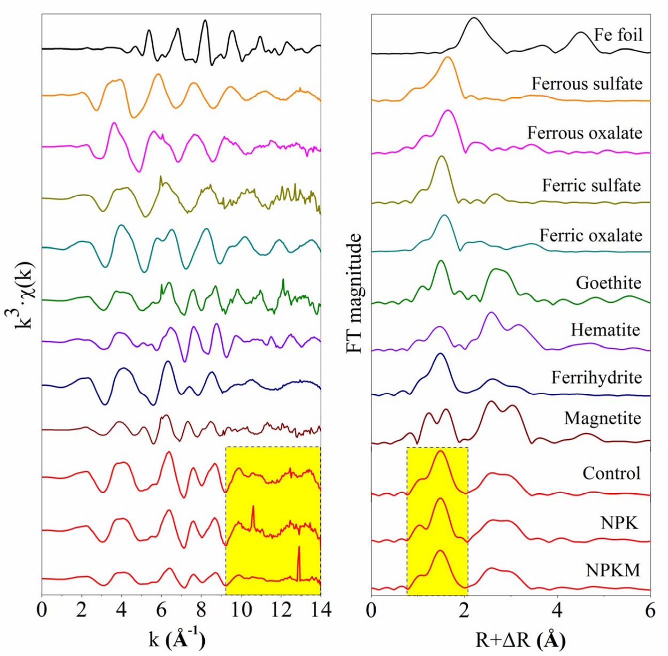

Fig. 5. Fe K-edge EXAFS (left) and Fourier transforms (right) of reference materials and soil colloids from three contrasting long-term (1990-2014) fertilization treatments