

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.

Anonymous Referee #1

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The very interesting objective of the manuscript (ms) is that NKP + manure fertilization enhances the formation of reactive Fe and Al nano-minerals that may contribute to the soil carbon stabilization. The soil samples were collected from a 24 years fertilization field experiment and were compared to a control soil and a NPK fertilized soil. The authors applied a set of modern investigation techniques like NanoSIMS, HRTEM, SEAD, XANES, EXAFS and XPS to characterize the fractionated soil colloids. It remains however unclear in the ms if all these techniques used and the measured data are neces-

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sary for the achieved results and their rather poor discussion and the conclusion. The authors referred to some more and less relevant references in the discussion instead of a comprehensive interpretation of the results. In fact, this is a rather methodological paper and the (new) mechanistic insights are lacking.

Overall, some fundamental information about the manure application and SOC stocks could be included in the ms and useful for the discussion. (See also my comments below) The authors can find this information e.g. in a recent review paper by Maillard and Angers, 2014.

Manure addition enhanced the org C binding of AI and Fe nano-minerals which was also shown in the previous papers of the authors like in Wen et al. 2014 a and b. The authors should clearly present what was achieved in that papers and what is really new in the ms besides the combination of applied modern investigation techniques.

Title: What are interactive characteristics? What about the other methods used in the manuscript?

Introduction: The authors should add a paragraph about the effect of fertilization practices on SOM development of soils, especially about the effect of manure!

Line 115: ferrihydrite and allophanes.

2.6 EXAFS and XANES: details of the data processing are missing. How was the data fitting procedure? How were they normalized and in which interval? How many single scans were used?... criteria for the best fit?

Results: Line 207: significantly 217-219: the findings of SAED measurements are similar to the finding of Wen et al, 2014 paper. Why were the SAED experiments needed? What are the new knowledge and the benefit of these results for the ms?

Line 224: Fig. 1 and S2: not really clear what the authors want to demonstrate with the HRTEM and NMR (!!) spectra Line 230: What is the relevance of Fig. S2 and Table S2 for 12C- rich or less rich ROIs? Table S2 is about metal composition! See also line

232 Line 233: why is the area of percentage similar for the Control and the NPKM?

Lines 238-239: These results are expected. See paper Maillard and Angers, 2014. Line 241: present

Lines 245-251: the XANES results about Fe oxide species are interesting! But see my comments about EXAFS.

Line 252-253: see my comments for lines 238-239, these results are expected!

EXAFS results (lines 254-279): The authors confirm the findings of XANES. Why were both EXAFS and/or XANES measurements needed? What is the benefit of the EXAFS or/and XANES results?

XPS: (lines 280-285): interesting results! Can a direct uptake of manure be distinguished?

4.1. The discussion about the increased concentration of reactive AI and Fe minerals is missing in the chapter (the authors only cite previous works about this). Also a discussion of the org C content and composition is missing in lines 301-306.

4.2 : a discussion about the possible mechanism of the formation of reactive AI and Fe mineral is missing. It is further unclear why both XANES and EXAFS results are necessary and useful for the discussion.

The author included in the Discussion part some recent papers and their statements and repeated the advantages of the used methods but this did not result in a comprehensive discussion about the formation of reactive Fe and AI minerals and their role for the SOC stabilization.

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