

Interactive comment on “New highlights on phytolith structure and occluded carbon location: 3-D X-ray microscopy and NanoSIMS results” by A. Alexandre et al.

Anonymous Referee #1

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Alexandre et al. have conducted interesting research to better understand the distribution of occluded carbon within phytoliths. A surge of recent research has pointed to the significance of this carbon pool for the global C cycle, but discrepancies between studies and applied methodologies have been the cause of diverse conclusions regarding its importance, and regarding its paleo-environmental meaning.

I agree with the authors that they provide an interesting research approach to this topic, with a combined usage of 3-D X-ray microscopy and Nano-scale Secondary Ion 10 Mass Spectrometry (NanoSIMS). The authors provide interesting data on the distribution of C within the silica structure of the phytoliths, and the existence of an organic template that participates in the phytolith formation, and as such contributes to

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the occluded carbon in the phytoliths.

However, I do not completely agree with the authors that they provide a big step forward in the understanding of big discrepancies in the phytC pool. These discrepancies have been linked to different location of carbon in the phytolith structure, e.g. in cavities and in holes on the surface of the phytolith. Authors using microwave digestion to remove organic matrices have claimed that underestimates result from using aggressive dissolution protocols for isolating the phytoliths (see this paper and references therein).

As I understand from the results and discussion in this paper, cavities and holes have been filled with air and/or epoxy resin during the applied procedures, making it impossible to quantify or qualify a cavity related carbon pool. While the authors acknowledge this throughout the paper, they still conclude that they provide new discussion material for these discrepancies, to re-assess the paleo-environmental meaning of the phytC. I think this conclusion is overly strong: the authors should refrain more to the conclusions they can perfectly draw from their research, on the distribution of C within the silica structure. They should provide discussion on how this approach can be improved to allow better understanding of cavity related carbon pools, rather than current, not-fully-supported, generalities like “These findings provide strong bases necessary to further characterize the nature, occlusion process, accessibility and origin of phytC. They also should help to reappraise the significance of phytC in the global C cycle and reassess the paleo-environmental meaning of phytolith features observed by NL microscopy”.

Further, the authors should better describe the amount of microscopical analyses they have performed, and whether conclusions could be generalized among all observations. It is unclear currently how many biomass replicates were studied (see also more detailed comments)

Detailed comments

- The authors mention in the introduction that aggressive chemical methods could re-

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move some phytC hypothesized to exist on the surface of phytoliths. How can they test whether phytC persists in holes on the phytolith surface, as earlier hypothesized, if they used aggressive extraction methods? On page 14707, the authors mention such cavities open to the surface, filled with air. If the cavities connect to the surface, can epoxy resin be incorporated in the cavities? The authors even mention this in the last part of the results section. How can they then discuss that they can quantify potential Si in cavities by comparing fossil to recent phytoliths? Can the C from the cavities not have been lost during the extraction?

- It is currently impossible to assess how many microscope pictures were taken, and how representative the samples from the *Triticum durum* were. How representative are the microscopical analysis for the sampled leaves? Why was this plant species chosen? How many leaves were used for the phytolith extraction? How many replicates of extraction? How many replicate pictures? Was their variability among observed patterns across (assumed) replicates?

Minor comments

14706, line 13: wide, not width?

14707, line 15: the structure

14700, Line 24: take up.

14701, line 5: Upon plant decay, or when plants decay

14701, line 25: remove bracket

14701, line 26: taken up

14703, line 2: measurements

Interactive comment on Biogeosciences Discuss., 11, 14699, 2014.

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