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Interactive comment on "Possible source of ancient carbon in phytolith concentrates from harvested grasses" *by* G. M. Santos et al.

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We thank the referee for his comments. Concerning the fact that the 14C data shown in this paper has been published elsewhere, we made sure that this information was acknowledged in the current paper. The text on p332 and 333 twice references Santos et al (2010a), and the captions for Tables 1 and 2 on pages 352 and 353 both include the phrase "Table adapted from Santos et al. (2010a)". More importantly, that study was quite different from the present one: it focused on the use of fine silica powder as a blank material to evaluate phytolith chemical extractions, and also attempted to obtain reproducible 14C results on replicate phytC (carbon occluded within phytoliths) samples produced from large pools of phytoliths extracted from soils and harvested living grasses using established protocols. Note that neither the phytolith chemical pro-

cedural blank assessment nor the reproducibility checks on 14C of phytC were ever attempted in previous studies. When 14C results of phytC from topsoil and living grasses were inexplicably old (though bulk material from the same plants gave contemporary 14C values), the focus of the study shifted to checking for sources of exogenous C contaminants. None was found, indicating that the 14C results were not an artifact of sample preparation or measurement.

What is new in the current paper is that we performed Scanning Electron Microscope studies coupled with Energy Dispersive Spectrometer analyses (SEM-EDS) using splits of the phytroliths from living plants that were previously 14C dated. Those images display the presence of organic matter (OM) outside the biosilica structure (i.e. not occluded within the phytolith) that, due to its recalcitrant properties, survived the phytolith extraction protocol which uses strong acids to oxidize and thereby remove the OM. This fact alone illustrates the inefficiency of the current phytolith extraction protocols cited in the manuscript, and suggests that the use of those protocols for 14C dating has intrinsic problems, undetected in previous studies.

Another aspect of this question, explored in detail for the first time in the current paper, is the likely association of the presence of OM residues on phytoliths concentrates with the fact that plants do not photosynthesize all the carbon found within their tissues. Literature surveyed in this paper shows that some of the carbon within bulk plant matter is taken up from soil as organic carbon through nitrogen assimilation (amino acids or proteins) and as soil dissolved inorganic carbon (DIC). As pointed out above, some OM within living plants is refractory and survives the phytolith extraction procedure, and we hypothesize that this fraction contains old soil carbon that contributes to the old phytC ages. Whether it is occluded in the phytoliths as well remains an open question. However, as isotopic analyses using phytoliths has been widely used in variety of applications (e.g. paleoclimate reconstructions, paleo-atmospheric CO2 records and archaeological research), we feel that raising the issue in this paper is a significant contribution on its own.

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To summarize, Santos et al (2010a) presented details of the methodology behind the measurements shown here in Tables 1 and 2, whereas the primary aim of this paper is to make the case that OM from soil has contributed to biasing the 14C ages of phytoliths extracted from recently harvested plants, and that by inference, essentially all phytolith 14C dates from previous investigations are suspect. This case is buttressed by work by Parr and coworkers (Parr et al. 2001; and Parr 2002) that demonstrates the presence of OM residues in phytolith concentrates extracted with three different procedures (dry ashing, heavy liquid floatation, and microwave digestion). We have cited this work at the end of the first paragraph in Section 5, to corroborate our findings on the impurity of the current extraction protocols.

Interactive comment on Biogeosciences Discuss., 9, 329, 2012.