

***Interactive comment on* “Comment on: “Possible source of ancient carbon in phytolith concentrates from harvested grasses” by G. M. Santos et al. (2012)” by L. A. Sullivan and J. F. Parr**

**R. Evett (Referee)**

revett@sonic.net

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General Comments

The technique of radiocarbon dating occluded carbon in opal phytoliths has been utilized sporadically since the pioneering work of Wilding (1967). In theory, because phytoliths are often abundant and more resistant to degradation than other forms of organic matter, particularly in grasslands and tropical forests, phytolith dating should be a valuable tool helping paleoecologists to better understand paleoenvironmental dynamics. In practice, researchers who have tried to incorporate phytolith dating into their studies have spent considerable time trying to explain anomalous dates, so the

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technique has not been widely applied. As is the case for many aspects of phytolith research, the technique has been applied before basic questions concerning phytolith radiocarbon dating, such as ascertaining that phytolith carbon from living plants produced modern dates, were properly tested. Prior to Santos et al. (2010), who found radiocarbon dates for phytoliths from modern grasses were up to several thousand years old, researchers assumed phytolith occluded carbon originated from photosynthesis. Santos et al. (2012a) published a review paper addressing this anomaly, summarizing the scant phytolith radiocarbon data available (including several unpublished sources) and proposed the hypothesis that at least some of the carbon in phytoliths is not of photosynthetic origin, but rather comes from older carbon in the soil taken up through the roots. Sullivan and Parr have authored a comment in reply to this paper, supplying additional data suggesting that the Santos et al. (2012a) hypothesis based on root uptake of old carbon may not withstand closer scrutiny.

Sullivan and Parr wrote their comment because they believe their research was misused by Santos et al. (2012a) to make two points. First, Santos et al. (2012a) used only the upper two of a sequence of 12 dates from a mature bamboo grove litter profile dated by Sullivan and Parr (2008) in an unpublished report to buttress their assertion that the few researchers who have dated modern phytoliths have found they were older than expected. They failed to mention that the remaining phytolith dates from lower in the profile were modern or near-modern. Sullivan and Parr believe these dates were omitted because they do not support the old soil carbon hypothesis. Second, Santos et al. (2012a), using EDS combined with SEM, asserted that all phytolith extraction protocols examined in their labs were inadequately pure for radiocarbon dating due to contamination from extraneous, non-occluded organic matter remaining in the extracts, and suggested this was likely true for other protocols they examined in the literature. Parr, who pioneered the microwave digestion extraction approach (Parr et al. (2001), is justifiably upset that Santos et al. deemed his extractions impure, not through SEM-EDS testing that was used for other approaches, but through questionable visual identification of extraneous organic matter in his published SEM photographs. The last part

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of the comment suggests that overly vigorous purification of external organic matter from phytolith extracts most likely removes some of the occluded carbon from within the phytolith, a valid concern.

Santos et al. (2012b) have recently replied to this comment. Their rebuttal is well-reasoned based on existing knowledge, but difficult to evaluate due to lack of data. It must be remembered that understanding the issue of old phytolith carbon is very early in the scientific process. Santos et al. (2012a) have proposed the outline of a hypothesis, based on a poorly-defined mechanism with very limited data, to explain a highly puzzling phenomenon. They have considered alternative hypotheses and deemed them less plausible. Sullivan and Parr, while offering a small amount of additional data, have attacked this hypothesis as lacking a plausible mechanism but not presented an alternative hypothesis in their comment. Until more data are available through further experimentation, this debate is almost completely based on speculation. Fortunately, many of the experiments required to understand the phenomenon are relatively straightforward and currently being performed by the Santos research team, so we can look forward to more substantial future discussions based increasingly on data rather than speculation.

Because Sullivan and Parr's comment is justified and based largely on their own work, supplying new data and a dissenting perspective, it is a useful addition to the phytolith carbon debate. I recommend publication, pending the very minor revisions discussed below.

## Specific Comments

Page 13775, line 12 – The term 'carbon fractionation mechanism' suggests the authors are referring to carbon isotope fractionation (a valid but implausible alternative hypothesis), when the intended meaning, based on both on line 6 on the same page and further discussion on P. 13776, lines 7-9, is carbon partitioning mechanism. Unless the authors are indeed referring to carbon isotope fractionation, which would require

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further explanation, the word ‘fractionation’ should be changed to ‘partitioning’.

Page 13778, lines 25 and 27 – Unless the authors are arguing for the carbon isotope fractionation hypothesis, which Santos et al. clearly are not, ‘fractionation/partitioning’ should be changed to ‘partitioning’.

#### Technical Corrections

Page 13776, lines 16-17 – The sentence within the quotation marks is a paraphrase, not a direct quote from Santos et al. (2012). The quotation marks should be removed and a comma placed between ‘literature’ and ‘that’, as well as (1996) and ‘and’.

Page 13780, line 34 – commas should be placed after each author’s last name.

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