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From radiocarbon analysis to interpretation: A comment on "Phytolith Radiocarbon Dating in Archaeological and Paleoecological Research: A Case Study of Phytoliths from Modern Neotropical Plants and a Review of the Previous Dating Evidence", Journal of Archaeological Science (2015), doi: 10.1016/j.jas.2015.06.002." by Dolores R. Piperno

Guaciara M. Santos [a, *], Anne Alexandre [b], Christine A. Prior [c]

[a] Earth System Science, University of California, Irvine, B321 Croul Hall, Irvine CA 92697-3100, USA
[b] CEREGE, UMR7330, CNRS-Aix-Marseille Université, Aix En Provence, France
[c] Rafter Radiocarbon Lab, National Isotope Centre, GNS Science, Lower Hutt, New Zealand

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ABSTRACT

The paper "Phytolith Radiocarbon Dating in Archaeological and Paleoecological Research: A Case Study of Phytoliths from Modern Neotropical Plants and a Review of the Previous Dating Evidence" by Dolores R. Piperno presents radiocarbon analysis of phytoliths from modern Neotropical plants collected between 1964 and 2013. The analyses presented were intended to rebut the emerging hypothesis that invokes root-plant uptake, transport and reallocation of soil organic carbon into phytoliths that has been recently put forward as an explanation for the anomalous radiocarbon (^{14}C) ages (of hundreds to thousands of years old) reported for modern grass phytoliths in Santos et al. (2010a, 2012a,b). We believe that the results presented in Piperno (2015) lack methodological rigor, mostly due to the absence of any procedural blank assessment, and that the attempts to disprove the hypothesis of uptake of soil organic matter (SOM) by phytoliths in Santos et al. (2012a) are not supported by a careful analysis. Rather than supporting the position that 100% of the carbon in phytoliths is of photosynthetic origin, which allows the use of phytolith carbon (or phytC) as a dating tool, the analysis of ^{14}C in phytoliths from modern Neotropical plants presented in the study shows that the ^{14}C ages are strongly affected by other sources of carbon. In this comment, we carefully reassess the ^{14}C results in phytoliths from modern Neotropical plants presented in Piperno (2015) in the context of the ^{14}C bomb-pulse methodology, SOM ages and turnover rates, and offer an alternative interpretation of the experimental results.

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* Corresponding author.
E-mail address: gdossant@uci.edu (G.M. Santos).

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1. Introduction

It is generally assumed that the source of phytC is atmospheric CO_2 fixed by the plant via photosynthesis (Wilding, 1967; Kelly et al., 1991; Raven, 2003; Carter, 2009; Piperno, 2006). It follows from this assumption that its ^{14}C signature can be used as a dating tool. However intuitively appealing this assumption might be, it cannot be accepted as scientific fact without adequate testing. Before asserting that phytolith ages from archaeological contexts are coeval with their sedimentary context (or expected ages), the assumption of a 100% photosynthetic source for the phytC must be validated by testing known-year post bomb specimens.

Piperno (2015) recently addressed anomalously old ^{14}C ages obtained for the carbon occluded in phytoliths extracted from living vegetation reported in Santos et al. (2010a, 2012a). Similar discrepancies have been reported previously in a scientific report to the Australian Institute of Nuclear Science and Engineering (e.g., Sullivan et al., 2008). In Santos et al. (2010a), the ^{14}C-AMS results were obtained from phytolith concentrates extracted from plants of known age harvested at two sites in USA and France, using an extraction protocol (Kelly et al., 1991) similar to that performed in several labs. Lab contaminants were systematically evaluated and a procedural blank obtained for each of them. In Sullivan et al. (2008) ^{14}C signatures were obtained from phytoliths extracted from harvested leaves of bamboo and the underlying litter layers, with the intention of reproducing the ^{14}C bomb-peak. Harvested leaves produced an age of 3.5 ka yrs BP, recently senesced leaves 1.9 ka yrs BP, and the litter ^{14}C results averaged ~100pMC (percent Modern Carbon) – significantly lower than the values expected from a bomb-peak signature. This dataset was published in Sullivan and Parr (2013) and discussed in an interactive comment by Santos et al. (2012b). Other researchers (Yin et al., 2014) have also reported unexpected low ^{14}C values, when trying to reproduce bomb ^{14}C signatures in phytoliths extracted from rice and millet harvested in 2011 and 2012.

In Piperno (2015), it is asserted that the anomalies reported in the direct ^{14}C dating of phytC are relatively recent and limited to extra-tropical regions. In order to refute the hypothesis proposed in Santos et al. (2010a, 2012a), ^{14}C signatures obtained from phytoliths extracted from modern Neotropical plants collected between 1964 and 2013 were reported. Since all but one of the ^{14}C ages exhibited a post-bomb ^{14}C signature (e.g, after 1955) it was claimed that no extraneous carbon biased the phytolith ^{14}C ages. Here, we present several reasons why this claim cannot be supported. The primary goal of this comment is to demonstrate how the ^{14}C bomb pulse methodology should be interpreted with respect to ^{14}C analysis of phytoliths. A secondary goal is to use that interpretation in order to draw attention to the implications and consequences of using phytC as a proxy of total plant carbon and in environmental reconstructions.

2. Radiocarbon bomb-pulse dating

Although there is abundant literature on the use of bomb-pulse dating, we briefly summarize the methodology below in order to clarify some common misperceptions.

Radiocarbon is a naturally occurring radioisotope produced in the atmosphere by cosmic rays. Radiocarbon is oxidized to carbon dioxide (CO_2) and enters the food chain through photosynthesis. Consequently all living organisms contain ^{14}C and continue to take in ^{14}C as long as they are alive. Once an organism dies or its parts (tree rings, for example) stop assimilating ^{14}C, the time-specific isotopic ratio of atmospheric $^{14}CO_2$ during the year of tissue formation is preserved. Atmospheric nuclear weapons testing in the late 1950s and early 1960s almost doubled the amount of atmospheric ^{14}C. After the banning of atmospheric nuclear testing in 1963, subsequent ^{14}C exchange with the ocean and land reservoirs, and dilution by fossil fuel emissions (Suess, 1955; Levin, 1997) caused the excess of atmospheric ^{14}C to gradually decrease. The penetration of atmospheric ^{14}C into terrestrial ecosystems has also led to a non-uniform labeling of soil profiles (Trumbore, 2000; Torn et al., 2009), with a reversal towards older pre-bomb ages below 10, 20 or >50 cm depth (Telles et al., 2003).

Almost 60 years of high resolution atmospheric $^{14}CO_2$ observations of the spike and subsequent decline at multiple locations in both the northern and southern hemispheres (Currie et al., 2011; Levin et al., 2013; Huan et al., 2013), have allowed researchers to use bomb ^{14}C as a very precise tracer. The ^{14}C content of terrestrial organic materials can be compared to the ^{14}C content of the atmospheric $^{14}CO_2$ bomb pulse as a way to: a) reveal modern forgeries (Caforio et al., 2014); b) identify poaching (Uno et al., 2013); c) validate the annual growth patterns in tropical tree species (Westbrook et al., 2006; Andreu-Hayles et al., 2015); d) determine birth- and/or death-age of subjects in forensic cases (Wild et al., 2000; Lynnerup et al., 2010); as well as e) verify sources of ^{14}C in organic materials (such as phytC).

Whether expressed as Fraction modern Carbon (FmC) or percent Modern Carbon (pMC) – where pMC = FmC × 100 – the carbon content of materials with ^{14}C signatures similar to those of atmospheric CO_2 after 1955 is generally referred as "modern" for simplicity. However, the usage of the term can create confusion. The mismatch between post-bomb or "modern" ^{14}C results for materials of known single calendar ages and the atmospheric ^{14}C content for that year can imply that the material measured contains a mixture of ^{14}C of different atmospheric calendar ages from the pre- and post-bomb periods. While pre-bomb ^{14}C values range from 0 to ≈99.9 pMC (equivalent to > 50ka yrs BP to 1955 calendar year, respectively), the 60 last years of the bomb-period also show large variations from 100 to approximately 180 pMC. As a result, small offsets in pMC within the bomb peak period are extremely significant and permit the identification of ^{14}C from different sources within organic materials. Similarly, when ^{14}C offsets between the true and measured ages occur during the pre-bomb period they also correspond to chronological offsets. For example, for each 1% of ^{14}C-free added, the expected age would be skewed by ~80 ^{14}C years towards older values, regardless of the true age of the sample (Aitken, 1990; Wood, 2015).

3. Comment on methods and the need for an experimental control

In order to compare the efficiency of different oxidizing agents for phytolith extraction, sulfuric acid (H_2SO_4) and a nitric acid/potassium chlorate mixture ($HNO_3/KClO_3$) were used in Piperno's work on duplicates. To ensure that all organic material was removed from phytolith surfaces, a second treatment was performed for additional hours. For ^{14}C analyses the phytolith extracts were combusted with tin to elevate the combustion temperature to 1400–1500 °C and therefore ensure phytolith melting. This is described as a necessary step according in Piperno and Stothert (2003), although this step has been shown to be unnecessary by other investigations (Table 2 in Santos et al., 2010a) who have shown that phytoliths combusted at 900 °C could be recovered and recombusted to verify combustion efficiency as well as background levels. The absence of CO_2 during recombustion confirmed that the combustion of phytoliths at 900 °C without the addition of tin reached completion without the addition of unnecessary catalysts, which can be potential sources of carbon contaminates (Ramsey and Humm, 2000). Whether or not higher combustion temperatures can be shown to improve combustion efficiency, the rest of

Table 1
Reproduction of the dataset in Piperno (2015). The averaged values for the pMC expected for each calendar year, the pMC offset between measured and expected, the estimated amount of %C added and the equivalent pre-bomb age offset in years is also provided. The age offset was calculated based on the 1%^{14}C-free to 80yrs relationship as per Aitken (1990) and Wood (2015).

Plant	Source	Processing method	Calendar year	pMC^{14}C-AMS	Beta Lab#	pMC expected	pMC offset	Amount of %C added	Age offset (yrs)
Zea mays	AASI	Nitric acid	1990–91	103.3	360,361	115.2	11.9	10.3	825
Zea mays	AASI	Sulfuric acid	1990–91	106.7	360,362	115.2	8.5	7.4	589
Cucurbita ecuadorensis	near Santo Domingo, Ecuador	Sulfuric acid	1995	114.2	360,359	112.0	−2.2	2.0	157
Cucurbita ecuadorensis	near Santo Domingo, Ecuador	Nitric acid	1995	114.4	306,082	112.0	−2.4	2.1	171
Cucurbita ficifolia	Chiriqui, Panama	Nitric acid	1995	101.8	368,019	112.0	10.2	9.1	729
Hirtella americana#1[a]	Fort Clayton, Panama	Nitric acid	1964	81.5[a]	360,360	180.0	98.5	54.7	4376
Hirtella americana#2	Albrook, Panama	Nitric acid	2013	100.1	368,020	104.0	3.9	3.8	300
Socratea durissima	Barro Colorado Island, Panama	Nitric acid	1981	123.1	368,023	125.9	2.8	2.2	180

[a] pMC^{14}C-AMS was determined based on the^{14}C age of 1640 ± 30 yrs BP, as seen in Table 2 in Piperno (2015).

Table 2
Reproduction of the dataset in Rieser et al. (2007) poster (data also shown in Prior et al., 2005; as cited in Santos et al., 2010a). NZA-is the Rafter, New Zealand sample identifier. CRA correspond to a conventional radiocarbon age before calibration.

Sample ID	NZA	CRA	Calibrated age range (±2σ)	OSL ages (ka)
A 2.85 (R28849/7)	21,974	10,404 ± 70	12,648–12,039 BP	15.2 ± 2.3
	22,534[a]	18,069 ± 90[a]	21,902–20,935 BP[a]	
A 3.15 (R28849/8)	21,975	6238 ± 100	7311–6790 BP	14.9 ± 1.8
	22,535[a]	16,703 ± 85[a]	20,067–19,566 BP[a]	
A 3.40 (R28849/9)	21,976	9960 ± 75	11,688–11,195 BP	18.0 ± 1.5
	22,536[a]	19,510 ± 110[a]	23,731–22,688 BP[a]	

[a] Repeat analysis with revised chemical extraction of phytoliths.

the protocol described in Piperno (2015) requires more detailed documentation in order to assess its robustness. For example.

a) No evidence of the purity of phytolith concentrates, either by light microscope or SEM-EDX (Santos et al., 2012a; Corbineau et al., 2013) was provided. This is necessary to check that all organic matter from the living tissue was removed and that extraneous carbon was not added.
b) The absorptive properties of pure phytoliths with regard to atmospheric CO_2 were also not assessed (Mintz et al., 2009; Hatté et al., 2010; Santos et al., 2010a). Trapped atmospheric CO_2 on the surfaces of phytolith extracts can be removed by use of a low temperature combustion step. The absorptive properties of phytoliths with regard to the use of solvents were also not considered. Santos et al. (2010a) demonstrated that solvent treatments applied to phytoliths bias their ^{14}C values (UCIAMS# 39,672 and −45447; Table 2 in Santos et al., 2010a). Nevertheless, Piperno (2015) suggests its use (first paragraph of Results section) without any evaluation of the effects on phytoliths concentrates, and consequently phytC ^{14}C.
c) No information is provided on phytolith concentrations by mass, amounts combusted, or yields from combustion and graphitization, all of which are necessary to assess the efficiency of the phytolith extraction protocol and ^{14}C sample processing. This is especially relevant, considering that the amount of C within pure phytoliths is on the order of 0.1–0.3% (Santos et al., 2010a). Several hundreds of mg of phytoliths have to be combusted in order to produce graphite samples of ≫ 0.1mgC for a measurement. A consequence of such small samples is that special attention must be applied to handle the small targets processed, the spectrometer measurements and inherent uncertainties. Greater uncertainties are generally linked to the combination of lower statistics (associated with low ion beam currents during spectrometer measurements, see Santos et al., 2007a,b) and background corrections from the addition of exogenous carbon during full sample processing (which should be determined by measuring a procedural blank, Santos et al., 2010b).
d) Most importantly, no procedural blank has been reported for any of the chemical extractions described above, and not for the carbon added during Accelerator Mass Spectrometry (AMS) sample processing. High chemical blanks (relative to sample size) might become a major contributor to the inaccuracy in the ^{14}C ages obtained and to overall uncertainties. To illustrate the importance of this issue, we note that dedicated research and explicit information on producing and measuring quality sub-milligram targets for ^{14}C-AMS measurements has been published by a number of authors (Santos et al., 2007a,b; Santos et a. 2010a; De Rooij et al., 2010; Fahrni et al., 2010; Wacker et al., 2013; Ruff et al., 2007). Neither this research nor any of these factors were discussed by Piperno (2015).

4. Comment on phytolith ^{14}C data analysis and interpretation

In Fig. 1, we reproduce the Neotropical phytC ^{14}C values reported in Piperno (2015) plotted against the ^{14}C bomb curve, except for the anomalous result associated with the 1964 calendar year (Tables 1 and 2 of Piperno (2015)). Although Panama and Ecuador are located across the Inter Tropical Convergence Zone and are therefore mostly within North Hemisphere zones 2 and 3 (NH Zone 2 and 3 – according Hua et al., 2013), after 1980 all ^{14}C timescale curves overlap so just one ^{14}C calibration curve (NH Zone 2) is plotted

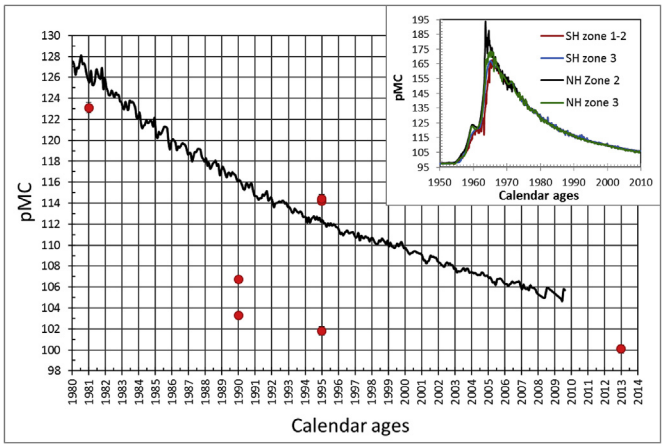

Fig. 1. Changes in atmospheric ^{14}C with time since 1950 are shown in the upper panel (solid lines). In this panel the time-scale calibration curves are split into zones, according to the conventions shown in Hua et al. (2013) to illustrate that most of the differences within the ^{14}C timescale curves across the northern and southern hemispheres falls between 1950 and 1980, while after 1980 all curves overlap. The phytC ^{14}C values of Piperno (2015) (red circles) are plotted in the lower panel against the Northern Hemisphere Zone 2 (NH Zone 2), except for the phytC ^{14}C value associated with the 1964 calendar year (discussed in the text). The uncertainties, as error bars, are smaller than the symbols in most cases. Radiocarbon values are expressed as percent Modern Carbon (pMC). (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article).

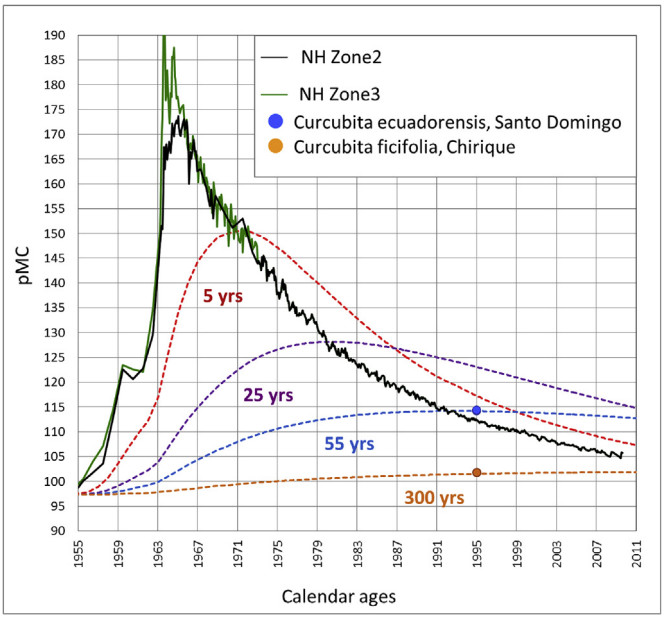

Fig. 2. pM^{14}C signatures from the Northern Hemisphere atmospheric ^{14}C timescale series (NH zone 2 and 3; Hua et al., 2013) and hypothetical SOM turnover times from a single/steady C pool reservoir. The phytC ^{14}C ages of *Cucurbita ecuadorensis* (blue circle) and *Cucurbita ficifolia* (red circle) in Piperno (2015) are also shown. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article).

together with the Neotropical phytC ^{14}C values. Regardless of the species tested, all pMC values appear depleted by ~3–12 pMC, except the duplicate values associated with phytolith concentrates from material collected near Santo Domingo Ecuador (Table 1 in this comment) for which ^{14}C is enriched by > 2 pMC. Assuming that these offsets are not a product of laboratory contamination, they are abnormal in a ^{14}C bomb-pulse dating perspective where ^{14}C annual signatures in each of the last 60 years can be separated, especially if they are from C pools expected to be 100% photosynthetic.

It was explained in Piperno (2015) that the bomb curve was not used to directly compare phytC pMC results with the calendar years of plant collection because local environmental variabilities can cause significant deviations from available bomb curve databases (details in small print under Table 2). However, such offsets have not been observed in Panamanian tree species *Hymenaea Courbaril* measured by Westbrook et al. (2006) that were obtained from an urbanized area near the city of David, Panama (8° 26′ N, 82° 26′ W). Even when carbonaceous resinous compounds from previous years were incompletely removed from the tree rings by chemical pretreatment, only the consecutive ^{14}C signals recorded in the tree rings between 1955 and 1964 were affected, while the rings between 1965 and 1997 were in complete agreement with the Northern Hemisphere ^{14}C atmospheric time scale. We regard the explanation of local environmental variability provided by the author to account for the pMC offset to be inadequate as it implies local CO_2 variations that are much larger than expected.

a) Anthropogenic CO_2 contributions due to the burning of fossil fuels (coal, petroleum and natural gas) can indeed significantly deplete atmospheric $^{14}CO_2$. Consequently, regional-scale ^{14}C fossil fuel maps have been generated to show spikes around the globe, or to monitor CO_2 emissions mitigation efforts. However, even close to large urban areas in the USA, biomass samples are ^{14}C depleted by only a few decades (Hsueh et al., 2007). In Los Angeles, one of the largest urbanized areas in Southern California, USA, the depletions in tree rings reached ≤5 pMC (Fig. 3 in Djuricin et al., 2012), whereas larger depletions require very close proximity to the fossil fuel sources (Riley et al., 2008). Note that North America is the second largest fossil-fuel CO_2 emitting region of the world, behind Asia with 1.70 billion tons of C emitted in 2008. In contrast, Ecuador (9.74×10^{-3} billion tons of C) and Panama (2.64×10^{-3} billion tons of C) ranked 71st and 100th, respectively, in CO_2 emissions from fossil-fuel burning, cement production, and gas flaring (Boden et al., 2011). Consequently, urban CO_2 emissions cannot possibly explain the widespread and anomalous ^{14}C depletions presented in Fig. 1. Proximity to active volcanic areas can also result in highly depleted ^{14}C values (Evans et al., 2010). However, since the ^{14}C data presented in Piperno (2015) come from plants collected between 1964 and 2013, and from different environmental settings, the possibility of a substantial volcanic influence cannot be evoked.

b) For the two samples collected near Santo Domingo, Ecuador (Beta- 360,359 and - 306,082), a positive source is required to explain the ^{14}C enrichment of ~2 pMC. In Piperno (2015; small print under Table 2), it is suggested that the ^{14}C of the plant tissue (and consequently phytC ^{14}C) was biased by frequent fires during the dry season. However a long duration and massive release of enriched $^{14}CO_2$ by fire (e.g. presumably stored in old forest biomass and top soil layers) would be required to skew those results, a mechanism that is not yet established. Moreover, a biogenic boost of ^{14}C higher than 1 pMC associated with forest proximity (native or commercial) and/or frequent use of biofuels (such as ethanol) has not yet been observed in any of the bomb pulse tree-ring ^{14}C datasets from the Western Southern Hemisphere Pantropical regions, including Panama (Westbrook et al., 2006), Brazil (Santos et al., 2015), Bolivia (Andreu-Hayle et al., 2015) and Argentina (Hadad et al., 2015). Heavy canopy vegetation effects (e.g., fixation of recycled CO_2

respired from soils produced by microbial decay of detrital carbon) are also unlikely, as they produce very small ^{14}C shifts (Trumbore, 2009). Unless the plants were collected from nearby a nuclear power plant (Wang et al., 2012; for example), a positive ^{14}C shift of this magnitude is improbable. In order to assess the origin of an apparent ^{14}C age offset of this magnitude in plant tissue, the ^{14}C of the tissue itself should have been analyzed for direct comparison to phytC ^{14}C.

c) In order to explain the anomalous thousands of years old age result obtained for the *H. americana* #1 (Beta-360,360) sample collected in 1964 (Tables 1 and 2, Piperno, 2015), Piperno states that shortly after the plant collection the material was sprayed with, or dipped in, pesticide of fossil fuel composition by the herbarium. However, a mass balance calculation (Table 1 of the present comment) indicates that for a sample harvested in 1964 to yield an ^{14}C age of 1640 ± 30 yrs BP, >50% of the graphite target measured by ^{14}C-AMS (and therefore phytC) must be of fossil-fuel origin (^{14}C-free or pMC = 0). This sample was subjected to a nitric acid/potassium chlorate mixture ($HNO_3/KClO_3$) extraction of several hours. It is hard to believe that the pesticide could still represent >50% of the phytC sample measured by ^{14}C-AMS, once the phytoliths have been fully extracted by a somewhat longer chemical treatment and when phytC is presumably completely occluded (and therefore, protected) within the silicate. If the ^{14}C of the tissue of origin had been analyzed for direct comparison to the phytC ^{14}C, this anomalous discrepancy could have been properly addressed.

d) The ^{14}C analysis of leaf phytoliths extracted from *H. americana* tree #2 (Beta-368,020) and collected in 2013, were used to evaluate the anomalous age of #1. Piperno states that the sample gave a post-bomb date "as expected". However, in 2013 the bomb-curve had reached the 104 pMC mark (based on the CO_2 monitoring station in Point Barrow, Alaska, Xaiomei Xu, Per. Communication). Therefore, the ^{14}C signature of this sample is also skewed, as it is showing an offset of ~4 pMC (Fig. 1, Table 1 of this comment).

In summary, all ^{14}C results for the post-bomb Neotropical samples with known year of collection reported in Piperno (2015) differ by > 2 to ~12 pMC from the expected ^{14}C atmospheric signatures. The *Hirtella americana* #1 phytoliths sample shows an even greater difference. Its ^{14}C signature is offset by 98.5pMC, since a plant growing in 1964 should measure ~180pMC. Such differences represent significant deviations from the expected ^{14}C values even when the combined uncertainties are taken into account (Fig. 1). They are also in significant disagreement with the ^{14}C time-scales obtained from a Panamanian tree ring/^{14}C-AMS dataset reported in Westbrook et al. (2006), which should have recorded the effects of some of the local atmospheric CO_2 anomalies invoked by Piperno (2015). In an effort to reconcile the discrepancies, Piperno speculates that CO_2 fixed from both fossil fuel emissions and fires must be present in plant tissue, or in the case of the anomalous result of phtC ^{14}C *Hirtella americana* #1, contamination by pesticide. Unfortunately, no proof that any of these events has occurred has been provided, such as measuring the corresponding bulk plant tissue by ^{14}C-AMS. We find it interesting that the offset range reported in Piperno (2015), excluding the *H. americana* #1, is similar to the one reported in Yin et al. (2014). They report offsets of ~3–13 pMC (see detailed information in supplementary material provided by the authors) that have been acknowledged as anomalous (see statements in the introduction and discussion of Piperno (2015)).

We suggest two explanations for these anomalous values. First, they are inaccurate because some unexpected source of exogenous carbon (old and/or young) was added during sample processing which was not accounted for due to the lack of processing blank(s) during phytolith extractions and AMS sample processing. In Table 1, we calculate the amounts of %C needed to skew the ^{14}C signatures and the associated ^{14}C age offsets. If these were due to the addition of ^{14}C-free carbon, such contributions would translate to ^{14}C age offsets between 157 and 4376 pre-bomb yrs. A second explanation could be that the plants took in small amounts of soil carbon through the roots (including post-bomb signatures), which were later allocated within phytoliths (the Santos et al., 2012a hypothesis). Santos et al., 2012a also point out the possible effects of incomplete digestion. If miniscule amounts of weathered-resistant SOM are already stored in plant tissue, upon digestion this oxidation-resistant OM tends to be left behind and therefore proportionally increases the transported soil-C residue in the phytolith concentrates. These remnants would be detected if SEM-EDX had been used to evaluate the purity of the phytolith concentrates (Corbineau et al., 2013).

5. Difference between SOM turnover time and ages - implications for phytC

A careful reading of Piperno (2015) seems to present a sequence of contradictory statements with regard to SOM age and turnover rates. In the first paragraph of the discussion section 4.1, it is stated that ^{14}C dates of 1250 BP to >2000 BP for soil depths between 12 and 20 cm have been found in Brazil, Peru, and Ecuador supporting the carbon persistence of plant remains in the tropics. Much later in the same section, it is also stated that "… the most resistant soil carbon fraction in northeast Brazilian soil was only 100 years old, one-tenth that of temperate soils (Chao and Holm, 1997)", and "… that tropical soils typically have faster SOM turnover rates than those in temperate biomes (e.g., Trumbore, 1993)" in order to reinforce the idea that old SOM uptake by plant roots in the tropics is unlikely. We would suggest that first of all, SOM fractions are highly heterogeneous among ecosystems and their vertical ^{14}C ages vary widely between locations, even in the tropics. For instance, Fig. 2 in Telles et al. (2003) shows several ^{14}C SOM profiles for a large array of Amazonian tropical forest soils, some with an uneven dispersal of bomb atmospheric ^{14}C with reversing patterns towards older pre-bomb ages reaching as much as 9.6 ka yrs at 200 cm in depth. Second, SOM turnover rates are generally defined as the carbon fluxes in and out of the C pool. Consequently, the mean residence time (MRT) of carbon in soils can be quite different than the individual ^{14}C ages of the large array of organic compounds comprising the SOM pool (Torn et al., 2009).

In Fig. 2, we show the predicted values of ^{14}C for a homogeneous, steady state reservoir with different turnover times. To illustrate that the phytC ^{14}C ages can reflect those of a mixture of atmospheric $^{14}CO_2$ and SOM, we also plotted the 1995 calendar age samples of *Cucurbita ecuadorensis* (Beta- 360,359 and − 306,082) and the *Cucurbita ficifolia* (Beta-368,020). Although we do not have the ^{14}C ages of the SOM or refractory fractions of the soils where those plants (and consequently phytC's) came from for a more precise comparison, it is quite likely that the carbon taken up by roots and reallocated into phytoliths of Santo Domingo (*Cucurbita ecuadorensis*; Beta-360,359 and − 306,082) came from a faster turnover-time SOM pool with many carbon fractions bearing post-bomb ^{14}C-enriched signatures (e.g. from previous decades). For the Chirique site (*Cucurbita ficifolia*; Beta-368,020) the phytC ^{14}C result falls on a plot of 300 yrs turnover time. This possibly implies that this site contains a much slower time scale (millennia) SOM pool (Torn et al., 2009), which would potentially negatively affect the phytC ^{14}C ages towards older values. Although the accuracy of these estimates cannot be evaluated without a thorough investigation of

the soil pools and their multiply-aged compounds, a SOM-derived C contribution to phytC still can be inferred from the ^{14}C results for post-bomb Neotropical Plants reported in Piperno (2015). Moreover, because the C in phytC may be from mixtures of labile to recalcitrant C pools with different chemical resistances to oxidation, its ^{14}C signature might be affected by the method used to extract the phytoliths (Santos et al., 2010a, Yin et al., 2014). The duplicate *Cucurbita ecuadorensis* (Beta- 360,359 and - 306,082) specimens processed in sulfuric and nitric yielded the same phytC ^{14}C pMC signatures within uncertainties, while those of *Z. mays* did not by ~3 pMC (Beta-360,361 and −360,362; Table 1 of this comment). If a sample comes from a C pool that is 100% homogeneous, measurements from different fractions should always yield similar ^{14}C values, independent of the method used during sample processing. This is demonstrated in Fernandez et al. (2015).

6. Interpretations of fossil and present phytC ^{14}C data

In Piperno (2015) it is stated that most attempts at phytolith dating and comparisons with independent dating techniques have been successful. The statement is supported by a series of references previously discussed in Santos et al. (2010a). To assert that at least some of these examples describing acceptable phytolith studies have been misinterpreted, we reproduce below the passages and datasets of some references cited in Piperno (2015).

The first reference is Wilding (1967). This paper reported the first attempt to directly date phytC by ^{14}C methodology and a date of 13,300 ± 450 years BP was obtained (I-2277). Unfortunately, in Wilding's words "… it was anticipated that the radiocarbon age of opal isolated from Warsaw soil would be between 1000 and 1500 years before the present … " He later states that "additional work is underway to understand the apparent anomaly between the anticipated and obtained carbon dates … " Wilding suggested that the 10 times greater than expected age was due to preferential oxidation of younger phytoliths and added: "Upon oxidation, such a phenomenon would favor preservation of older carbon occlusions at the expense of younger ones, and thus may account in part for the older carbon date obtained". Thus, "Preferential oxidation would not affect the validity of the date as an estimate of the minimum age of the valley train sediments. It would, however, preclude the use of such dates to reconstruct ecologically the major period of grass vegetation at a particular site." Note the importance of the last sentence. For this reason, we conclude that the work of Wilding (1967) cannot correctly be cited as a successful application of the radiocarbon dating of phytoliths, as further analyses were still pending.

Piperno also cites research by Rieser et al. (2007) as producing credible phytC ^{14}C results that match expected ages. In Table 2 of the present comment, phytolith ^{14}C results and Optically Stimulated Luminescence (OSL) ages of the associated Kawakawa tephra (New Zealand) produced by Rieser et al. (2007) in their poster, are presented. The first batch of phytoliths extracted at Victoria University and analyzed at the Rafter Radiocarbon laboratory returned unexpectedly young ages. After a more rigorous oxidation was applied, the phytC ^{14}C ages became older by at least 8ka yrs BP, and consequently older than the associated OSL ages (Table 2). While further OSL analyses on materials associated with the Kawakawa tephra from different sites (e.g. Schermer et al., 2009) suggested that the tephra could possibly be younger than previously reported, these revised phytC ^{14}C ages were still significantly younger than the accepted radiocarbon age of the Kawakawa tephra (25,360 ± 160 cal yr BP, Vandergoes et al., 2013). Because the phytC ^{14}C dates matched neither the OSL ages nor previously published radiocarbon ages for the tephra, the data from this study were never formally published as a paper, while further testing of phytolith extraction methods and dating continued. Regardless of the true age of the Kawakawa tephra, it is clear that the phytolith ages of the revised extractions reported in Rieser et al. (2007) are several thousand years older than the OSL ages of the surrounding sediments, contrary to what is implied in Piperno (2015).

Although Piperno (2015) acknowledges the abnormality of the phyt^{14}C ages of 3510 and 1865 BP for living and recently senesced bamboo leaves in Sullivan et al. (2008), the "modern" phyt^{14}C ages extracted from the leaf litter were presented as evidence that the phytoliths did not take up old SOM. Santos et al. (2012b) reanalyzed the Sullivan et al. (2008), Sullivan and Parr (2013) dataset using a corrected post bomb-pulse methodology and found age anomalies of at least 400 years towards older values. As highlighted in section 2, and in Table 1 (this comment) "modern" or post-bomb pMC does not automatically imply correct ^{14}C ages.

In Piperno (2015) it was also suggested that Santos et al. (2010a, 2012a) assumed that all phytoliths found in the uppermost few cm of soils should be of modern age. This is incorrect, as the investigators were certainly aware that there is always a mixture of old and new phytoliths even in the uppermost layers of soils (Alexandre et al., 1997, 2011). Santos et al. (2010a) pointed out large differences in the ^{14}C ages of SOM and phytC from the same soil layer (e.g. McClaran and Umlauf, 2000) and that these differences would have been even larger had a high purity extraction methodology been used. Other references cited by Piperno (2015) that echo similar problems have been extensively discussed in Santos et al. (2010a, 2012a,b), and although it is tempting to challenge them one by one again, these refutations will not be repeated here.

Attempts to resolve some of the difficulties with direct ^{14}C dating of phytoliths have prompted researchers to investigate other independent phytolith repositories. A post-bomb *Nothofagus truncata* phytolith extract yielded an anomalous ^{14}C age of 2152 ± 60 yrs BP (July 2007 NZA-28219, R29539, measurement performed by Dr. Prior at Rafter). In parallel, phytoliths from upper soil layers from the Sahelian zone, Senegal, Africa (April 2008 UCIAMS47170, S.27, measurement performed by Dr. Santos at UCI) returned a^{14}C age of 3750 ± 15 yrs BP. In this last example, inconsistency is found not just in the remarkably older age of the phytC topsoil layer, but also in the fact that the phytolith assemblage from S.27 mirrors modern vegetation, whereas 4000 years ago the vegetation in the same area was quite different (e.g. Alexandre et al., 1997). Because these phytC ^{14}C dates were clearly anomalous and failed to support the 100% photosynthetic carbon signal in phytC, they were also never formally published. However, they clearly highlight our concerns regarding the assessment of paleoenvironmental and paleodiet reconstructions based on geochemical analyses of phytC.

7. Conclusions

In our view, the post bomb results newly presented in Piperno (2015) were misinterpreted, as were most of the reinterpretations on the discussions in Santos et al. (2010a, 2012a, 2012b), both of which support the systematic offset in ^{14}C signature of phytC and probable extraneous soil carbon contribution to phytC.

We clarify the points raised by the author regarding her reinterpretations of ^{14}C phytolith dating in the literature, and reassess the ^{14}C results for phytoliths from modern Neotropical plants in the context of a standard ^{14}C bomb-pulse methodology. However, the lack of comparison between the ^{14}C signatures of phytC and the ^{14}C signatures of the plant of origin prevents further assessment of the hypothetical explanations provided by Piperno (2015) for the reported anomalous depleted and enriched phytC ^{14}C values. Moreover, since the quality of the presented ^{14}C results cannot be properly evaluated due to the lack of important information on phytolith purity, phytolith carbon recovery, and procedural blank

assessment, an exogenous source of C cannot be completely dismissed.

If the ^{14}C results presented in Piperno (2015) are correct, and are correctly interpreted, they add to the long list of phytoliths from modern plants with unexpectedly anomalous ^{14}C ages. They show ^{14}C signature offsets equivalent to 157 to 4376 pre-bomb years. This may imply that previous pre-bomb phytC ^{14}C from Neotropical regions of the world might be affected by SOM contributions to phytC, and therefore should be carefully and rigorously reappraised. Considering that ^{14}C analysis are more accessible than ever, and can be provided by multiple research and commercial facilities, we hope that this comment will help future scholars to be more critical and careful when examining radiocarbon data.

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Further readings

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