“Standard Evaluations of Bomb Curves and Age Calibrations Along with Consideration of Environmental and Biological Variability Show the Rigor of Phytolith Dates on Modern Neotropical Plants: Review of Comment by Santos, Alexandre, and Prior”
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Abstract

Santos et al. claim that a recent phytolith ^{14}C study by Piperno of Neotropical plants that grew during the post-bomb era provided anomalously old ages due to ^{14}C depletion. They argue the depletion source is likely old carbon in soils transported into plants via root uptake. Here I show: 1) their claims for anomalous ^{14}C depletions in phytoliths are unfounded because they fail to consider uncertainties created in the bomb curve from local and regional environmental variability and other factors shown to lead to bomb curve offsets in post-bomb ^{14}C study, 2) they error by not calibrating the phytolith dates, a standard procedure with post-bomb ^{14}C determinations, 3) they inexplicably consider an ancient (1640 $^{14}\text{Cyr B.P}$) age for one of the dated samples to be accurate when (a) it is known the sample was treated with substances made from fossil fuels that were not removed with the extraction process, and (b) the amount of radiocarbon dead carbon required to generate the ancient age from SOM is unreasonable, and 4) their theory that old soil carbon from root uptake is sequestered in phytoliths causing significant skews to phytolith ages is not supported by accumulated evidence from ancient, and now modern Neotropical contexts.

Keywords: Phytolith post-bomb ^{14}C dates, Standard evaluations of post-bomb dates, Inherent regional and biological variability affecting bomb curves.

1. Introduction

Piperno (2015) reported post-bomb phytolith ages from a variety of modern Neotropical plants, an expected result that was consistent with their collection dates after 1950. Santos et al. (2015; e.g., pg. 7) claim that “widespread and anomalous ^{14}C depletions” occur in the dates that call into question phytolith dating accuracy, and that one of the samples contained an extraneous source of modern carbon adversely affecting its age. As background to this discussion, the atmospheric bomb testing that occurred during the 1950s and early 60s resulted in a large increase in ^{14}C in air as bioavailable $^{14}\text{CO}_2$ that reached a peak in about 1963 after the Test Ban Treaty was signed, and then started to decline. Plants growing after 1955, therefore, have elevated ^{14}C signatures, reflected in percent modern carbon (pMC) values greater than 100. Santos et al. (2015) (hereafter Santos et al.) draw their claims of ^{14}C depletions in the dated phytoliths from plotting the percent modern carbon (pMC) values of the specimens against what is called the ^{14}C bomb curve that displays changes in atmospheric ^{14}C over the post-bomb period (Fig. 1). Santos et al. further argue for what they consider to be problematical phytolith dates by calculating a total age offset in years (the Age offset column in their Table 1) through multiplying the difference between the phytolith pMC and the predicted bomb curve pMC for that collection year by a 1% ^{14}C free to 80yr. relationship.

As will be shown here, evaluating the post-bomb ^{14}C ages in these ways is inappropriate for a number of reasons. Santos et al. take available bomb curve data as “absolute”, disregarding internal error within the data itself and localized and regional variability (Darden Hood, Beta Analytic Inc., pers. comm. 2015). Furthermore, the 1% ^{14}C free to 80yr relationship, based

strictly on using the Libby half-life to calculate radiocarbon ages, assumes that amounts of fully radiocarbon-dead carbon have contaminated samples and is applicable to pre-bomb but not these post-bomb results because ^{14}C years and calendar years while comparable for pre-bomb dating, are significantly different for the post-bomb interval (Darden Hood pers. comm. 2015 and further explanation below). Santos et al. also problematically failed to use a standard approach for assessing a specimen's pMC vis a vis its collection date, through calibration of the specimen's age using its pMC with established calibration tools for post-bomb ages (e.g., Reimer et al., 2004).

Here I: 1) discuss in more detail the pMC values of the phytoliths in Piperno (2015) and compare them with values from other plant materials, including leaves and tree rings, in routine post-bomb analyses of other investigators, 2) provide calibrated ages for the phytoliths, showing they routinely produce reasonable ages and results consistent within the error limitations one can expect from bomb carbon prediction data, 3) provide additional details of the environmental contexts and biological characteristics of the plants studied, showing these were not given the attention by Santos et al. that was warranted, and 4) discuss how the Santos et al. theory of old carbon uptake and subsequent segregation in phytoliths from the Neotropics and perhaps other regions is currently poorly supported by a number of different lines of evidence.

2. Bomb Curve Use, Environmental Differences, and the Samples Studied

It is well known in radiocarbon study that bomb curves are generalized per very large global regions (atmospheric CO_2 and ^{14}C concentrations are sub-hemispheric averages and only latitude confined), and subjective with regard to any single sample. Therefore, perfection should not be expected when attempting to correlate a post-bomb age to a year of collection; inherent local and regional variations are likely to exist anywhere and it's entirely reasonable to assume at the

outset a 1-2 pMC offset either way in any location (Darden Hood, pers. comm. 2015). It is well-
 documented that measureable pMC depletion occurs in urban areas due to fossil fuel emissions
 from various sources (e.g., Hseuh et al., 2007; Pataki et al., 2010). Both living corn in areas of
 China and living grasses along busy streets in Japan have been measured with a 5 pMC
 depletion, and in northern California annual forest fires also produce significant amounts of older
 carbon from the internal rings of trees (Darden Hood, pers. comm. 2015). Areas near volcanoes
 are known to have depleted atmospheres (e.g., Chatters et al., 1969; Cook et al., 2001; Evans et
 al., 2010). There is currently no universally accepted correction for inherent local variability in
 bomb curve data as there is, for example, for the marine reservoir effect (Darden Hood, pers.
 comm. 2015). Moreover, the 1% ^{14}C free to 80yr. relationship used by Santos et al. to compute
 their age offsets for the phytolith dates (their Table 1) does not apply to the interpretations of the
 phytolith results. The relationship can apply when assessing a pre-bomb radiocarbon age using
 the Libby half-life equation; in contrast, when interpreting post-bomb ages the pMC is compared
 directly to the bomb curve calendar year data without use of the Libby half-life (Darden Hood,
 pers. comm.). The stark differences obtained can be easily seen, as pMC went, for example, from
 pMC 116 to 112 in five years according to bomb curve data (Fig. 1), not the age difference of
 320 years ($4 \text{ pMC} \times 80$) as would be computed by Santos et al for the pMC difference. The use
 of this relationship by Santos et al. also ignores again localized and regional variability in bomb
 curve data, as it assumes that all phytolith pMC offsets from the bomb curve are due to
 extraneous, dead carbon.

Samples studied in Piperno (2015) were from a range of different environments in Ecuador,
 Panama, and the United States. The Santos et al. claim that a single tree ring datum from near
 David, Panama (Westbrook et al., 2004) proves that environmental variables had no influence on

the samples studied or inadequately account for observed pMC depletions is untenable, including with regard to the three plants from Panama tested. David is a small city (population 140,000) located in western Panama on the Pacific coast. Westbrook et al. give the collecting locality as “near the city of David”, presumably outside the city, which quickly becomes rural (Piperno, personal observations), and do not state the collection area is “urbanized” as claimed by Santos et al. Furthermore, it is 400 km from the Panama City area, where the *Hirtella americana* specimens came from and ^{14}C depletion from air pollution is an issue, and 60 km from the Chiriqui uplands where *Cucurbita ficifolia* was collected. The latter locale sits on the slopes of a volcano that is emitting gases depleted in ^{14}C . Fires in old growth forest growing on volcanic soils also occur there, and because tropical trees live for hundreds of years carbon injected into the air from burnt wood or aerobic decay would be old. These and other issues will be addressed in detail for each plant studied.

2.1 Cucurbita ecuadorensis, Collected February, 1995

This is a spontaneously-growing fruit collected along the route between the towns of 14 de Mayo and Santo Domingo, Ecuador (Olaf Holm, pers. comm. 1995)¹. Two samples from it were processed separately with nitric and sulfuric acid. Results

Footnote 1. I did not state in Piperno (2015) as Santos et al (pg. 7) claim, that this particular sample was “biased by frequent fires during the dry season”. Rather, in the notes to Table 2 in Piperno (2015) where all of the results were given the general statement was made that “The bomb curve calibration was not used to directly compare pMC results with the dates of plant collection since local environmental variability, such as fires set annually during the dry season or proximity to urban centers, can cause significant deviation from available bomb curve databases (Darden Hood, personal communication, 2015).” No specification of which samples were subject to fires or proximity to urban settings was made; this information is now given in this paper.

were the same in each, with pMCs of 114.4 and 114.2, respectively (Table 1). The pMC for 1995 plotted directly from the bomb curve is about 112, a 2.2 to 2.4 pMC difference. Santos et al. claim on the basis of this small difference that a source of extraneous enriched carbon biased the sample and a 114 pMC is readily explained only if a power plant was nearby. In doing so, they

disregard the inherent uncertainties of available bomb curve data and erroneously take them as an absolute (Darden Hood pers. comm. 2015). Furthermore, when the phytolith date is evaluated by determining the calibrated age using its sample pMC and a standard calibration tool for post-bomb determinations, CALIBomb (<http://calib.qub.ac.uk/CALIBomb/>), the result is a two-sigma age range of AD 1958-1994.46, with a high probability of accuracy in the range segment 1990-1994.46 (see Table 1 notes for a detailed explanation of date calibrations and Fig. 2). The calibrated age is within less than a year of the collection date and therefore of entirely acceptable accuracy in post-bomb study (below), further refuting the Santos et al. claim the date is skewed.

It is worth further pointing out to investigators not familiar with bomb curve interpretations and evaluations of post-bomb dates how both the pMC relationship to the bomb curve for *C. ecuadorensis* considered problematic by Santos et al. and the calibrated age range with respect to the sample collection date compare with other bomb pulse studies of plant materials. Ehleringer et al. (2011) examined whole coca (*Erthroxylum coca*) leaves from fields in rural settings in Colombia, Ecuador, Peru, and Bolivia where traditional cultivation practices were used. When compared with the bomb curve, specimens provided pMC differences similar to or greater than *C. ecuadorensis*, with values in coca both depleted (falling below the bomb curve line) and enriched (above the line) (Table 2). Calibrated ages for coca routinely yielded ranges the same as and even further removed from collection dates than *C. ecuadorensis* (Table 2). All were considered unremarkable by Ehleringer et al. due to bomb curve uncertainties. Studies of tree ring specimens and cotton seeds from Arizona were similar in these regards (Goodsite et al., 2001) (Table 2). Ehleringer et al. found their results to be of significant utility for constructing a regression for predicting the age of specimens for forensic use by Drug Enforcement agencies.

I also note that “age offsets” that Santos et al. would calculate and obviously were not a part of Ehleringer et al.’s, Goodsite et al.’s interpretations or of the bomb curve literature at large (a few examples are Garnett and Stevenson, 2004; Reimer et al., 2004; James et al., 2010; Pataki et al., 2010; Buchholst, 2012; Georgiadou, et al., 2013; Uno et al., 2013; Caforio et al., 2014; Leifeld et al., 2015) were similar to or larger in coca and tree rings than in *C. ecuadorensis* (Table 2). Also, coca leaves collected on the same day from the same country showed pMC differences (e.g., the Peruvian June 1, 1994 samples, Table 2). These results re-inforce the point that ^{14}C depletions/enrichments and pMC offsets from the curve that Santos et al. assert to be problematic in phytolith dating for *C. ecuadorensis* and other plants analyzed in Piperno (2015) (below) routinely result from bomb curve generalities and uncertainties it and local and regional environmental variability create, even in non-industrial settings.

2.2. *Socratea durissima*, Collected in 1981 and *Hirtella americana*, Collected in 1964 and November 22, 2013

Socratea durissima (name recently changed to *S. exhorrida*) and *Hirtella americana* were to my knowledge the first living woody species studied for phytolith ^{14}C . The former is an understory palm species in tropical forest and has a life-span of at least a few decades. It was collected from Barro Colorado Island (BCI), Panama, a 1600 ha forest reserve in Gatun Lake in the Panama. Forest burning or clearing haven’t occurred at BCI for hundreds of years (Piperno, 1990), thus the sample comes from a local environment that is presently undisturbed and natural. The pMC for *S. durissima* phytoliths is 123.1 and the bomb curve-predicted pMC is about 125. The calibration yields a two-sigma age range of AD 1959-1985, overlapping the collection date, with increasing probabilities of age in the 1982-1985 range with use of NH3 and SH3 curve extensions. This again reflects how the Panama atmosphere is influenced by intense mixing from

ITCZ air masses moving annually from the south (Table 1 notes for further explanation and Westbrook et al., 2004). Once again, the collection year is within a year of a calibrated range and within the prediction capability of the bomb curve, contradicting the Santos et al claim the phytolith age is skewed and anomalous.

With regard to this sample and the question of whether root uptake of old carbon from soil organic matter biases phytolith dates (further discussed below), a number of factors are evident. It is known that palms in humid Neotropical forest tend to be shallow rooted, often considerably less than a meter in depth (H. Joseph Wright, pers. comm., 2015) and unlikely, then, to have much root penetration into deeper soil depths where most old SOM is found. The root/SOM factor is further minimized in *S. durissima* because it is a “stilt palm” with aerial roots and the great proportion of its root mass above ground. In light of the valid phytolith age from *S. durissima* and known associated root conditions, root uptake of old SOM can be ruled out as an influencing factor.

H. americana is a canopy tree with a typical life-span of a few hundred years. Phytoliths from two samples were dated, a specimen (#1) collected in Albrook, Panama and deposited in the STRI herbarium by another investigator in 1964, and another (#2) collected by Piperno’s associates in November, 2013 in Clayton, Panama. As was explained in Piperno (2015), the leaf of #1, which provided an age of 1640 ± 30 ^{14}C yr B.P., was treated in the herbarium by spraying with and/or dipping it in the then-used pesticides/fungicides lauryl pentachlorophenate and paradichlorobenzene. The two are chlorinated hydrocarbons and rich sources of radiocarbon-dead carbon from fossil fuels. Naphthalene, another product made from fossil fuels, was also placed around herbarium plants at that time. Because Santos et al. assume the processing procedure used (nitric acid with potassium chlorate; see Piperno, 2015) would remove the

pesticides, they find it hard to believe they biased the sample. Basic chemistry disagrees. The substances are soluble in organic solvents such as acetone, benzene, and chloroform, not in the chemicals I used or in water (see, for example, the websites <http://chemyq.com/En/xz/xz12/116743xgcvj.htm> , <https://myorganicchemistry.wikispaces.com/Paradichlorobenzene> , and <http://www.chemicaland21.com/industrialchem/organic/P-DICHLOROBENZENE.htm> (note: P-dichlorobenzene is another name for paradichlorobenzene). The substances would have saturated epidermal tissue and phytoliths, which are epidermally-derived, and it is well-known how silica surfaces are particularly adsorbent, including to pesticides (e.g. Tertykh, 2004; Ramola et al., 2013). Santos et al. disregard the dead carbon contamination and use the bomb curve-predicted 180 pMC for 1964 along with the 1% ^{14}C free to 80yr. relationship to compute a 4376-year “age offset” for the sample. This clearly cannot be taken seriously. Furthermore, for SOM carbon to account for the observed depletion, 60% of the carbon in the phytoliths would have to come from radiocarbon dead SOM in an active Neotropical A horizon. Alternatively if the soil SOM was 5700 BP, 79% of the carbon in the phytoliths would have to come from SOM (Darden Hood pers. comm.2015). This is an unreasonable expectation in a tropical environment where turnover is rapid. (Santos et al. complain that analyses such as SEM EDX weren’t done on extracted phytoliths to examine specimen purity and that the study lacked “methodological rigor, mostly due to the absence of any procedural blank assessment”. The procedures used are established in Piperno’s laboratory and shown over a 30 year-plus period to provide clean preparations and suitable samples for ^{14}C study, including by SEM examination (Piperno and Stothert, 2003, Piperno, 2006). Extractions proceeded normally, organic matter was removed, and clean phytolith

samples with sufficient carbon for a standard AMS analysis--from about 190 to 330 micrograms--
 were graphitized by Beta Analytic. Validation of accuracy in the results prior to reporting was
 achieved through measurement of similar size internal reference standards including materials
 such as OXII, IAEA-C3, IAEA-C9, FIRI-I and internal secondary standards, and the accurate
 results on these reference standards provided suitable validation assessments for assigning
 accuracy to the AMS measurements on the phytoliths [Darden Hood, pers. comm. 2015]. High
 carbon content in the submitted samples (>190 micrograms) provided routine sample sizes for
 which AMS laboratory background was well-defined, and high ^{14}C contents (>100 pMC)
 minimized effect of background on the final results; thus, procedural blanks mentioned by Santos
 et al. would only relate to additional background which may have been introduced during
 phytolith extraction and preparation [Darden Hood, pers. comm. 2015]). The latter also should
 not have affected results since processing took place in a clean, closed laboratory with materials
 and chemicals suitable for ^{14}C study that do not introduce carbon [e.g., Piperno, 2006, 2015]).

A fresh sample #2 of *H. americana*, collected near the end of 2013 from next to a busy street
 just six km down the road from #1 in the same environmental context, is the only relevant one of
 this species to discuss. Predicting pMC values from the bomb curve and age calibrations are
 subject to greater uncertainty, in part because the curve has not yet been adjusted for consensus
 atmospheric CO_2 values after 2010 and the sample pMC of 100.1 will cross the curve only on the
 up (older) side (Fig. 1), and also because when the pMC is close to 100 the lower limit of the +/-
 can extend into the time period before bomb-carbon onset (i.e. less than 100 pMC). In those
 cases, there is some probability for 18th, 19th, or 20th century antiquity when calibrating, as
 shown by the ranges for *H. americana* in Table 1.

Santos et al. assign a pMC of 104 for *H. americana* based on a 2013 value from a single monitoring station in Point Barrow, Alaska, a pristine environment high in the northern hemisphere often used as a clean air baseline comparative for ^{14}C depletion studies of urban polluted air (Pataki et al., 2010). It's unclear to begin with if this reference point can be used for the tropical location in question. A consensus has developed that the value at present (2015) is 102 pMC based on routine measurements made at a $^{14}\text{CO}_2$ monitoring station in The Netherlands (Lutjewad, Groningen), and it is not unreasonable to consider that inherent local effects on the *Hirtella* specimen could produce values as low as 101 pMC, consistent with its measured result of 100.1 (Darden Hood, pers. comm. 2015).

What's indeed clear is that the local environment for *H. americana* is far from pristine and may well have exerted measureable influences on pMC levels. The collecting locality is near the urban core of the Panama City metro area, by orders of magnitude the largest population concentration in the country (1.44 million--over half of all Panama), and clogged with traffic from numerous cars and buses. Among industrial activities, large construction projects demand high cement production. Smog forms most years and air pollution is a serious problem. Santos et al. dismissed out of hand air pollution as a factor, citing a statistic that Panama is 100th on the list of carbon-producing countries, and failing to realize most of that production comes from a single, densely populated area, Panama City (about 8000 people per sq. mile, rivaling Los Angeles). A more relevant statistic than given by Santos et al is that in metric tons per capita of CO_2 emissions, Panama emits more than Brazil and not much less than Mexico, both of the latter among the top ten overall emitters in the world (<http://data.worldbank.org/indicator/EN.ATM.CO2E.PC>). In sum then, in light of the ^{14}C depletions in both the air and plant samples documented from urban fossil-fuel emissions from

similar areas elsewhere (Hseuh et al., 2007; Riley et al., 2008; Pataki et al., 2010)—for example, from 2.5 to 5 pMC in Los Angeles and surrounding areas (Pataki et al., 2010)—this factor cannot be excluded from evaluations of this plant.

2.3 *C. ficifolia*, Collected in January, 1995

This specimen had a pMC of 101.8 and the bomb curve-predicted pMC for 1994-1995 is 112. The calibrated phytolith age is AD 1955-1956, thus any ageing effect is no greater than about 40 years. In this case, fossil carbon may have been introduced but not through the normal channels of respiration. Santos et al.'s statement that volcanic activity did not affect any of the samples is erroneous. The *ficifolia* fruit was collected about eight to 10 km northwest of the town of Boquete in highland Chiriqui, Panama (Thomas Andres, pers. comm. to Piperno, 1995 and 2015), an area that sits on the eastern flank of Volcán Baru, a large and ancient (0.5 Ma) stratovolcano that dominates the region. It erupted four times in the past 1600 years, the last being 400 to 500 years ago (Sherrod et al., 2007). These and other large eruptions during the Pleistocene and Holocene showered thick, 10-20 cm layers of tephra over a large area, including where the plant was collected, which is on the side of the volcano where Pleistocene lava domes and flow are identified and not more than 5-8 km from the domes (Fig. 8 in Sherrod et al., 2007). Hikers reaching the Baru dome complex with its seven craters describe steaming fumeroles (vents), hot springs, and bubbling mud pools, which continually emit gases with old carbon dioxide into the air and are signatures of active magma chambers.

Also, at least three strong seismic swarms (many volcano-derived tectonic events within a short period of time that often precede eruptions) lasting several weeks occurred in the area the past 50 years; in 1963, 1985, and 2006, and swarms are probably under-reported (Sherrod et al., 2007). Swarms are known to inject gases high in dead carbon dioxide from moving magma

beneath the volcano and deplete atmospheric ^{14}C levels, with effects lasting into subsequent years (Cook et al., 2001; Evans et al., 2010). Volcanoes dormant for hundreds of years can emit gases (Pasquier-Cardin et al., 1999). Emissions with depletion and ageing effects on vegetation can also take place in areas removed from those with visible gas emissions through diffuse degassing of the magma-derived tephra soils (e.g., Allard et al., 1991; Pasquier-Cardin et al., 1999; Soter, 2011).

Studies of CO_2 and ^{14}C in the area's air and ground in order to assess by how much these factors influence local vegetation ^{14}C ages have not been carried out. Nonetheless, a possible effect needs to be taken into consideration when evaluating this specimen, as the local conditions summarized above could easily cause the plant pMC and bomb curve differences. One more point here. Given the volcanic soils rich in highly ^{14}C -depleted and dead carbon the plant grew in, if a significant amount of soil carbon entered the plant via roots and was then sequestered in phytoliths, as expected in the Santos et al. scenario (below), wouldn't a date earlier than post-bomb be expected?

2.4. Zea maize, Collection Date Uncertain

An error was made when Piperno (2015) stated the maize that was analyzed arrived in Piperno's lab in 1990-91. It arrived later at a date and location uncertain from the United States, with the first record of it in the Piperno laboratory occurring in starch grain records compiled in 1997. Thus, the pMC recorded for the samples cannot be evaluated with respect to collection year and calibrations. The two samples analyzed on glumes and cupules combined from the same cob produced pMC values of 103.3 and 106.7. The local growing and post-collection curation conditions before it was sent to Panama are unknown and the lack of available information prevents us from commenting further. Considering these uncertainties, modern maize from

known contexts will be assessed in the near future for phytolith age. It is noted that each sample yielded a post-bomb age with a maximum too-old error of about 35-40 years or 10 to 15 years too young, assuming a collection year sometime between 1991 and 1997 (Table 1).

3. The Santos et al. Theory of Old Soil Carbon in Phytoliths

Santos et al. (2010, 2015) theorize a prominent reason for aberrantly old phytolith ages recorded by other investigators, including themselves, is that two different pools of carbon exist in phytoliths; one from photosynthesis and the other from old soil organic matter (SOM) taken up by roots. Comments on this theory were previously made by myself and others, and won't be repeated in detail here. Briefly, there is no argument that plants may uptake small amounts of soil carbon through their roots (e.g., Soter, 2011, Hodson, 2012, 2015). The real issue is to what extent it affects dates from phytoliths compared with other plant components. Because so much more carbon enters plant tissue via photosynthesis than through the xylem (vascular system), in order for the Santos et al. SOM theory to be viable, the SOM would have to be largely sequestered inside phytoliths, while the abundant photosynthetic source would seemingly have to be preferentially excluded from them (Hodson, 2012). No mechanism or evidence is known for such an effect, which is difficult to envision (Casey et al., 2003; Hodson, 2012, 2015). Furthermore, if any significant amount of old SOM were to be taken up and not sequestered in phytoliths, it would seem that radiocarbon analysis generally would be liable to too-old ages. The theory also doesn't fit well with phytolith ^{14}C results from Australia that Santos et al. argued were prone to an old SOM error (Sullivan and Parr, 2012; Piperno, 2015). Soter (2011) addressed old carbon uptake by plant roots with relation to possible errors in plant ^{14}C analysis generally. The very small quantities of carbon from roots in different plants studied, ranging

from about 0.1% to a few percent of the total plant carbon, resulted in an estimated maximum age error in a ^{14}C date on conventional materials of a few hundred years (Soter, 2011).

I noted previously that SOM is a complicated issue with many facets that may vary from one environmental context to another (Piperno, 2015), but there is little doubt that SOM is more quickly cycled in the warm, humid conditions of the tropics than in the temperate zone (e.g., Trumbore, 1993; Chao and Holm, 1997; Krull et al., 2002). It would seem, then, that any attention to this issue should proceed on a region-by-region basis. Finally, Santos et al. embark on a deeply speculative discussion of estimates for soil carbon turnover age in the collection areas of the plants discussed here, from which they derive their figure 2, admitting: “Although the accuracy of these estimates cannot be evaluated without a thorough investigation of the soil pools and their multiply-aged compounds..”, but then inferring in a circular argument the phytoliths must have contained old SOM. Indeed, their 303-313 year turnover time for *C. ficifolia* soils is inapplicable to the volcanic soil context.

4. Other Inconsistencies and Errors in Santos et al.

Apart from the above issues, there are a number of inconsistencies and inaccurate statements in Santos et al, including confusions and mis-quoting of discussions in Piperno (2015). Space constraints don’t permit addressing all of them. Among the notable examples, they begin their section 5 on page nine, titled “Difference between SOM turnover time and ages - implications for phytC”, with the following:

“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.”

In contrast, here is the complete sentence with the 1250 to > 2000 BP dates referred to by Santos et al.:

“¹⁴C dates on charcoal fragments ranging from 1250 BP to more than 2000 BP from the uppermost 12-20 cm of terrestrial soil in Brazil, Peru, and Ecuador and Ecuador (McMichael et al., 2012; Piperno et al., in press) further support the point that plant remains of that age that can survive for long periods are found in uppermost soil horizons.” (Piperno, 2015, pg. 3).

It’s clear the 1250 to >2000 BP dates were from *charcoal* found at that depth in the soils, not SOM, and a look at the sentences preceding it will make it obvious that I referred to the charcoal dates when discussing the subject of long-term *phytolith persistence* in soils and its effects on phytolith dates from upper soil horizons, not SOM survival; discussion of SOM and its survival took place much later in the paper. SOM and charcoal are different entities studied separately (charcoal is removed from soils before SOM analysis—e.g., Telles et al., 2003). The charcoal sentence was reproduced incompletely, taken out of context, and used to argue for something I did not, then to call my words contradictory. Another example occurs on Santos et al. pg. 5, where they state I suggested in Piperno (2015) using solvent treatments in phytolith dating procedures. The reader can compare for themselves what they say I say with what I said, which was in relation to the chemicals that would have been needed--discussed above in the *H. americana* #1 section— *if* removal of the pesticide substances from *H. americana* #1 were to be attempted. It is clear in Piperno (2015) that I did not attempt to remove them or advocate doing so, and have nowhere recommended for phytolith dating solvents employed in Santos et al. studies or others (organic solvents mentioned above), emphasizing, in fact, how they might bias phytolith ages (e.g., Piperno, 2006:94).²

Footnote 2. As another example of a confusing discussion, they claim a date of 3750 BP obtained on phytoliths from upper soils in Senegal isn’t reasonable because phytolith assemblages reflect modern vegetation when it’s known that 4000 years ago the vegetation in the area was very different, citing Alexandre et al. (1997). However, the latter paper discusses phytolith distributions in a 2m-deep soil sequence from the Dimonika area, Congo, quite distant from Senegal, and there is no indication the authors consider the record to be 4000 years old or reflective of vegetation change (e.g. they state, “From the top to the bottom of the profile, the circular rugous morphotype ..., a phytolith from dicotyledon stems... accounts for 72 to 90% of the phytoliths counted...[Alexandre et al. 1997:678]).

5. Summary and Conclusions

I showed here that phytoliths from known age plants collected during the post-bomb era routinely produce reasonable calendar ages when properly pretreated, correct use of available bomb-curve data is made, and relevant environmental and other uncertainties are considered. Detailed discussions here of a few years offset from published bomb curve data were made to clarify the oversights of Santos et al.'s interpretations, and, combined with other discussions here, are meant as well to provide for users of ^{14}C results that are reported to them, reasonable examples of how bomb pulse data are evaluated and interpreted, as phytolith dating moves forward in the future. I discuss multiple lines of evidence that phytolith dates were not significantly affected by SOM carbon. The real issue in phytolith AMS dating is explaining grossly too-old ages (several 100-1000s of years ^{14}C BP) obtained in other studies, prominently on recently harvested plants (Santos et al., 2010; Sullivan and Parr 2012). Furthermore, previous ^{14}C determinations obtained from archaeological and paleoecological contexts in the Neotropics were rigorously appraised by their comparison to associated charcoal, shell, and other ages (see Piperno, 2006, 2015; Piperno and Stothert, 2003). If, as Santos et al. insist on asserting, the phytoliths contained a source of old carbon that makes ^{14}C determinations unacceptable, anomalously old dates should have been obtained, not the dates that compared well with determinations on other materials long-used in carbon-14 study.

Obviously, old carbon present in the carbon reservoir would produce pMC values inconsistent with a known age for phytoliths and I demonstrate here sources of exogenous carbon that can affect the ^{14}C result, factors that apply to any radiocarbon-dated sample. However, evaluations of the Santos et al. SOM theory have identified a number of problems, and it surely

shouldn't be turned into a worldwide assumption when evaluating phytolith ages. As previously noted (Piperno, 2015), answering the questions that have come to light will involve investigating differences in processing procedures.

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Table 1. Percent Modern Carbon (pMC) and Calibrated Ages for the Plants Studied in Piperno, 2015

Specimen	Sample Year	Location	Phytolith pMC	Calibrated Phytolith Age (Two Sigma)
<i>Cucurbita ecuadorensis</i>	1995	near Santo Domingo Ecuador	114.4 ± 0.4 Beta-360359	SH Zone 3 extension [cal AD 1958.73 :cal AD 1959.13]0.132 [cal AD 1987.43 :cal AD 1987.46]0.002 [cal AD 1988.47 :cal AD 1988.57]0.010 [cal AD 1989.52 :cal AD 1989.53]0.001 [cal AD 1990.10 :cal AD 1994.46]0.856
<i>C. ecuadorensis</i>	1995	same fruit as above	114.2 ± 0.3 Beta-306082	Calibration same as above.
<i>Socratea durissima</i>	1981	BCI, Panama	123.1 ± 0.5 Beta-368023	NH Zone 2 extension [cal AD 1959.27 :cal AD 1961.67]0.571 [cal AD 1982.19 :cal AD 1982.58]0.024 [cal AD 1982.78 :cal AD 1984.15]0.393 [cal AD 1984.88 :cal AD 1985.00]0.012 NH Zone 3 extension [cal AD 1959.37 :cal AD 1960.06]0.138 [cal AD 1960.90 :cal AD 1961.79]0.226 [cal AD 1982.15 :cal AD 1985.02]0.637 SH Zone 3 extension [cal AD 1961.85 :cal AD 1962.72]0.114 [cal AD 1963.06 :cal AD 1963.25]0.024 [cal AD 1982.11 :cal AD 1985.21]0.861

Hirtella Americana #1 (Albrook, Panama, 1640 ± 30 B.P. Beta-360360, contained fossil fuel substances, see the text)

<i>Hirtella americana</i> #2	2013 Clayton, Panama	100.1 ± 0.4 pMC Beta-368020	NH2 extension [cal AD 1698.31 :cal AD 1722.03]0.091 [cal AD 1815.92 :cal AD 1834.01]0.060 [cal AD 1878.37 :cal AD 1915.97]0.511 [cal AD 1954.62 :cal AD 1955.97] 0.338
<i>Cucurbita ficifolia</i>	1995 Chiriqui, Panama	101.8 ± 0.4 Beta-368019	NH2 extension [cal AD 1955.52 :cal AD 1956.22]1.000
<i>Zea mays</i> Nitric acid	Collection date uncertain, see text	103.3 ± 0.3 Beta-360361	NH Zone 2 extension [cal AD 1955.96 :cal AD 1956.68]1.000
<i>Zea mays</i> Sulfuric acid	Collection date uncertain, see text	106.7 ± 0.3 Beta-360362	[cal AD 1956.99 :cal AD 1957.68]0.102 [cal AD 2003.04 :cal AD 2003.63]0.031 [cal AD 2003.84 :cal AD 2007.72]0.850 [cal AD 2008.50 :cal AD 2008.73]0.015 [cal AD 2009.50 :cal AD 2009.52]0.001

Notes: In way of explanation of post-bomb date calibrations using *C. ecuadorensis* as an example, a two-sigma calibrated age range was obtained from CALIBomb of AD 1958-1994.46. The SH3 zone was used for calibration following the zone boundaries delineated in <http://calib.qub.ac.uk/CALIBomb/>. The 0.132 and 0.856 after the first and last increments of the two-sigma range represent what's called "probability density functions", which is the likelihood of the correct date to be in that range, in this case 13.2 and 85.6 percent, respectively. The two-sigma range starting in the 1950s and ending decades later for this and other samples in Tables 1 and 2 reflects the fact that a pMC may intersect the bomb curve twice--one on the side going up (the pMC reached a maximum value of about 193 in 1963) and one on the side going down (for the *C. ecuadorensis* bomb curve graph see Fig. 2; F14C units are converted to pMC by multiplying by 100.). The probability density function on the calibration contributes to assessment of which date range is most likely to be correct, as also does knowledge of the specimen collection year when available, as in this study. The calibrated ranges may also

be in smaller increments than in pre-bomb dating, as seen in *C. ecuadorensis* and other plants here, due to rapid changes in atmospheric ^{14}C in the post-bomb era. All samples in Tables 1 and 2 were calibrated using the intcal13 calibration data set in CALIBomb.

The Panamanian collection areas lie near the boundary of NH zone 2 and NH zone 3 to its south and close to SH zone 3 (see <http://calib.qub.ac.uk/CALIBomb/>). A study of Panamanian tree rings (Westbrook et al., 2004) discusses how a Panama record will be expected to more closely resemble the southern hemisphere atmospheric record due to the annual movement from south to north over Panama of the intertropical convergence zone (ITCZ) and the intense mixing and recycling of air masses associated with the movements. Some of the Panama dates were therefore calibrated with the NH2, NH3 and SH zone 3 bomb curve extensions to compare and contrast. When different extensions aren't provided for Panamanian dates, the calibrated ranges differed very little. The maize samples were calibrated with the NH Zone 2 extension.

Table 2. Examples of Post-Bomb Results from Other Studies

Specimen	Sample Date	Location	Sample pMC	Calibrated Age (Two Sigma)				
Coca leaf	January 1, 2000	Colombia	107.7 ¹ .	NH Zone 2 Extension [cal AD 1957.26 :cal AD 1957.88]0.076 [cal AD 2001.08 :cal AD 2001.47]0.045 [cal AD 2001.78 :cal AD 2005.18]0.866 [cal AD 2005.52 :cal AD 2005.78]0.013				
				NH Zone 3 [cal AD 1957.81 :cal AD 1958.09]0.051 [cal AD 2001.07 :cal AD 2001.48]0.062 [cal AD 2001.76 :cal AD 2005.16]0.881 [cal AD 2005.64 :cal AD 2005.75]0.006				
				¹ . The bomb curve-predicted pMC is about 109.9 leading to a 168 year “age offset”, This and other age offsets in this Table were computed by Piperno for discussion purposes (see text) and were not a part of the interpretations of this and other studies referred to in this Table.				
				Coca leaf	October 18, 1993	Colombia	110.9 ² .	NH Zone 2 Extension [cal AD 1957.74 :cal AD 1958.38]0.049 [cal AD 1994.27 :cal AD 1994.28]0.000 [cal AD 1994.94 :cal AD 1994.96]0.001 [cal AD 1995.20 :cal AD 2000.06]0.949
								NH Zone 3 [cal AD 1958.14 :cal AD 1958.43]0.047 [cal AD 1995.21 :cal AD 2000.05]0.953

²The predicted pMC is about 113.5 leading to a 208 year age offset.

Coca leaf September 5, 1993 Colombia 111³

3. The predicted pMC is about 113.4 leading to a 200 year age offset.

NH Zone 2

[cal AD 1957.76 :cal AD 1958.43]0.054
[cal AD 1993.86 :cal AD 1993.88]0.001
[cal AD 1994.26 :cal AD 1994.32]0.003
[cal AD 1994.78 :cal AD 1999.23]0.924
[cal AD 1999.49 :cal AD 1999.88]0.018

Coca leaf June 1, 1994 Peru 110.3⁴.

⁴The predicted pMC is about 112.5 leading to a 176 year age offset.

NH Zone 2

[cal AD 1957.69 :cal AD 1958.25]0.046
[cal AD 1996.03 :cal AD 2000.69]0.954

Coca leaf June 1, 1994 Peru 113.1

NH Zone 2

[cal AD 1957.95 :cal AD 1958.71]0.069
[cal AD 1991.15 :cal AD 1991.24]0.006
[cal AD 1991.77 :cal AD 1995.12]0.898
[cal AD 1995.52 :cal AD 1995.82]0.027

Coca leaf August 10, 2007 Colombia 107⁵.

⁵The predicted pMC is about 106, leading to a 80 yr. age offset.

NH Zone 2

[cal AD 1957.12 :cal AD 1957.71]0.089
[cal AD 2002.26 :cal AD 2002.34]0.005
[cal AD 2002.88 :cal AD 2006.90]0.903
[cal AD 2007.10 :cal AD 2007.15]0.003

Douglas Fir Tree ring 1985 Arizona, USA 123.8⁶.

⁶The predicted pMC is about 121 leading to a 224 yr. age offset.

[cal AD 1959.26 :cal AD 1961.81]0.426
[cal AD 1980.18 :cal AD 1980.21]0.001
[cal AD 1980.92 :cal AD 1981.38]0.026
[cal AD 1981.58 :cal AD 1984.16]0.535
[cal AD 1984.68 :cal AD 1985.01]0.011

Douglas Fir Tree ring	1983	Arizona, USA	124.5 ⁷ .	[cal AD 1959.38 :cal AD 1960.82]0.172 [cal AD 1980.13 :cal AD 1980.27]0.012 [cal AD 1980.81 :cal AD 1984.11]0.733 [cal AD 1984.90 :cal AD 1984.99]0.007
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⁷The predicted pMC is about 123, leading to a 120 yr. age offset.

Cotton seed	1984	Arizona, USA	122.6	[cal AD 1959.18 :cal AD 1961.64]0.641 [cal AD 1982.50 :cal AD 1982.54]0.003 [cal AD 1982.82 :cal AD 1985.02]0.355 [cal AD 1985.52 :cal AD 1985.53]0.001
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Notes: Because it is unclear if Colombian coca samples were collected from within NH zone 2 or NH zone 3, some were calibrated with both bomb extensions for comparison; there was little difference between the two. The Peruvian coca sample was calibrated with the Southern Hemisphere Zone 1-2 extension, and tree ring and seed samples with NH Zone 2, as indicated on <http://calib.qub.ac.uk/CALIBomb/>. The coca leaf and tree ring values are from Table 4 of Ehrlenger et al., 2001 and Table 1 of Goodsite et al., 2011, respectively. Calibrations for coca, tree rings, and cotton seeds were carried out by Piperno.

Figure 1

Figure 2