

## ***Interactive comment on “High resolution <sup>14</sup>C bomb-peak dating and climate response analyses of subseasonal stable isotope signals in wood of the African baobab – A case study from Oman” by Franziska Slotta et al.***

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Dear Dr. Mazumdar, dear referees,

We gratefully acknowledge the efforts of Dr. Santos and Dr. Chakraborty for their comprehensive reviews of our manuscript. Their questions, criticism and thoughtful comments are very appreciated. In this response – prior to the editor’s decision - we provide additional input and perspective that make some of the referees’ concerns less pressing and may dispel others altogether. In the revised MS minor comments will

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thoroughly be considered, grammar and spelling mistakes corrected.

The most critical comments of the referees can be summarized three key points: 1) our paper does not discuss in sufficient detail all potential causes of the observed F<sup>14</sup>C values of tree-ring cellulose being significantly lower than in the corresponding atmospheric CO<sub>2</sub> for the period around the bomb peak. Referees question our arguments for significant tree-physiological causes and particularly criticize the lack of discussion of external reasons. 2) our methodological and analytical description is lacking detail, unclear, ambiguous or not up to the mark. 3) The stable isotope data, our analysis of climate-proxy relationships and the climatological interpretation has raised almost no interest of the referees at all.

If given the opportunity by the editor to revise our MS we will particularly focus on addressing these three issues. We will consider and incorporate the literature suggested by the referees. In view of this we will carefully re-write our methods section exemplifying that sample preparation and mass spectrometric analyses (IRMS and AMS) were performed in compliance with international standards and good scientific practice. In the discussion we will be taking into special consideration the potential external causes of <sup>14</sup>C change during the period of the bomb peak (e.g. fossil fuel burning, atmospheric circulation etc. as put forward by the referees). In the following, we outline the key changes we intend to incorporate into the revised manuscript to consider the individual key points raised by the referees.

Comments to Referee #1: General remarks

The paper presents annual <sup>14</sup>C data from an African baobab (*Adansonia digitata*) tree from Oman, for the interval AD 1941 to 2005. This work is important in that it provides a fairly detailed pre/post-bomb <sup>14</sup>C time-series for a region that has not yet being part of the atmospheric <sup>14</sup>C global compilation. This is actually one of the main goals of the manuscript. The authors have also improved the quality of the data set by providing intra-annual analyses of  $\delta^{13}\text{C}$  and  $\delta^{18}\text{O}$ , as well as F<sup>14</sup>C for the calendar years of 1962

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and 1963. Response: We thank the reviewer for considering this work as important for a region that is underrepresented in terms of 14C data.

While the high number of consecutive single tree-rings measured by radiocarbon allowed confirming the annual nature of the baobab species, a significant mismatch between the baobab F14C values and the post-bomb atmospheric curve NH3 was detected. This mismatch prompted an alternative explanation, i.e. mixed pool of slow-turnover non-structural carbon (NSC) into the structural ring cellulose fraction - a strong functional trait of parenchyma-rich tree species (maybe ?!). Response: We agree with this summarizing statement of our observations. However, we do not just explain the aberrant F14C values by incorporation of carbon from a mixed pool of slow and fast turnover of NSC. In the MS we propose an additional potential cause: namely the huge difference in longevity of wood forming plant cells: while parenchyma cells can live up to approx. 20 years, wood fibres live up only from a few weeks to a season. This means that parenchyma tissue can undergo changes in its 14C for over several years, whereas the 14C of fibre tissue is always assigned to a certain year. Since in baobabs parenchyma occurs not only as bands but is also diffusely distributed within a tree ring, varying proportions of parenchyma and fibers can cause variations in F14C of a tree ring that can be, to a certain extent, unrelated to the specific date of the tree ring. Please note, that baobabs are unique in this regard. To our knowledge, no other tree species shows similarly high parenchyma contents than baobabs (69-88%). Changes in the manuscript: We will rewrite introduction and discussion to make these two tree physiological aspects clearer. In addition, we will discuss in more detail potential external causes of the observed 14C trends (see responses to further comments below).

Comment: The Baobab terminal parenchyma bands F14C values presented here, definitely demonstrate that a large percent of the parenchyma in this tree species is relatively young, and as such, it provides valuable perspectives in the field of plant physiology. On the other hand, mixed carbon pools in putative structural ring cellulose fraction (in this case, slow-turnover NSC residue in holocellulose extracts) put into

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question the use of tree rings of this group of woody plant when reconstructing atmospheric 14C. Response: Thank you for supporting our conclusion that our F14F data points to future perspectives in plant physiological research (in particular on baobabs, which are widely distributed in Africa and potentially threatened by global change). Our data set contributes a fairly detailed pre/post-bomb 14C time-series for a region that has not yet being part of the atmospheric 14C global compilation. However, it was NOT the main purpose of our MS to reconstruct atmospheric 14C from this data set. As written in the introduction, we primarily intended to use the 14C bomb peak to validate the counting/dating of tree rings. As mentioned by the referee above, the unexpected significant mismatch between the baobab F14C values and the post-bomb atmospheric curve NH3 prompted for some reasonable interpretation. In this case we suggested and still suggest that it is from a mixed carbon pool in conjunction with the extraordinary high content and longevity of parenchyma tissue relative to short lived fibre tissue that constitute the tree rings of baobabs. Nonetheless, the referee is right. Tree species with such a high content of parenchyma should not be used for reconstructing atmospheric 14C. This raises particular issue for tropical regions, where tree angiosperm species show about 36% of parenchyma on average. In contrast, angiosperm tree species in temperate zones have a content of about 21%, only. Changes in the manuscript: We will rewrite introduction to clarify the original purpose of our 14C analyses and we will add and detail the aspects outlined above in the discussion of the revised MS.

Comment: I appreciate that in view of the perplexing results of the 14C data of the baobab tree rings, an alternative explanation should be considered. However, for the mixed pool NSC-ring cellulose assumption works, all other possible bias must be carefully ruled out. Response: Thank you very much for this valuable comment. This point, i.e. other potential causes for the observed bias, has also been raised by referee #2. Changes in the manuscript: As suggested, we will add a paragraph tackling other, external effects on atmospheric 14C to the MS and also rephrase the parts in the manuscript referring to this.

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Comment: Robust methodologies must be properly done and explained in detail, as well as the use of reference materials/internal standard, or equivalent (i.e. interlaboratory measurements), and the use of further chemical extractions. All of those are missing here. Response: Thank you very much for pointing to the lack of description of our methodology. This point has partly also been raised by referee #2. Changes to the manuscript: As suggested, we will carefully rewrite the methods section and explain in more detail the process of tree ring dissection, cellulose extraction and mass spectrometric analyses.

Comment: Given the absence of an independent benchmark, e.g. a short F14C sequence of consecutive single tree-rings from a non-parenchyma-rich woody plant in Oman, I cannot tell whether slow-turnover NSC detected in holocellulose extracts of baobab tree rings is a feasible explanation for the 14C offset observed here or not. For starters, 14C analysis of incomplete single tree rings (material that do not represent a full growing season) could contribute in 14C offsets (see specific comments/suggestions). Response: Good point. Unfortunately, project resources were limited and did not allow analyses of other tree species than baobabs. 14C analyses were done on the 2/3 of a tree ring. The last 1/3 including each terminal parenchyma band was discarded in order to minimize negative effects from long living parenchyma tissue. Since the transition from wood to the terminal parenchyma band cannot be determined precisely, we decided to also skip some of the wood. We believe that contamination from parenchyma causes larger bias than the seasonal effects. Baobab tree rings in Oman are largely formed between May and October each year; our samples from may represent the growing period from May to August or early September. Changes to the MS: We will exemplify our sampling strategy in more detail.

Comment: Furthermore, we had to keep in view that other factors must also play some role in those 14C offsets (atmospheric circulation and carbon dioxide from human activities, for example). Previous records across zones NH3, SH3 and SH1-2 are very scarce. Therefore, the possibility of multiple sources of air-14CO<sub>2</sub> influencing Oman

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should be discussed. Response: Yes, we admit that these factors have not been discussed in detail. This point has been raised by referee #2 as well. Changes to the MS: We will carefully add and discuss these aspects.

Comment: One cannot ignore the fact that during the assembly of the atmospheric post-AD 1950 14C global compilation by Hua et al. (2013) some datasets were disregarded due to its mismatches with other regional datasets. Therefore, a thorough evaluation of possible external effects should also be offered. Response: Yes, once more, we admit that these factors have not been discussed in detail. Changes to the MS: We will carefully add and discuss these aspects.

Comment: Finally, procedures described here need further explanations and details. Response: Yes, thank you for this comment. Changes to the MS: We will exemplify all our procedures in much more detail.

Comment: The result and discussion part is quite jumpy and very tricky to follow. It does not quite convey the ideas of the underlying assumption offered to explain the baobab tree F14C offsets. I recommend a complete re-organization of the manuscript, by focusing on placing the absolutely necessary data, figures and tables (for the purpose of the paper) in the main text. Response: Thank you for this comment. Apparently, there is some misunderstanding concerning the purpose of this paper. It is not ment to present a reconstruction of atmospheric 14C (see earlier response comments above).

Changes to the MS: We will rephrase the MS to make our intentions much clearer.

The stable isotope findings were not particularly striking. Although important, they are currently creating a lot of distraction. I strongly suggest moving them (most of its description, associated material and discussions) to a supplementary text or appendix. Response: The intra-annual stable isotope data, its climate related analysis and interpretation is of valuable interest to the dendro- and palaeoclimate community, which may not be the major audience of BG and the referee of this MS. In particular, stable iso-

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tope data points to distinct relations between intra-seasonal stable isotope patterns and pre- and post monsoon cyclones. Furthermore, tree-ring stable isotope records from regions like Oman are still scarce and knowledge about the climatic significance of baobab tree-ring parameters is important. According to her expertise, the referee feels distracted while focusing on the radiocarbon part, only. Note, there seems to be some misunderstanding concerning the purpose of this paper, which was not ment to present a reconstruction of atmospheric  $^{14}\text{C}$  (see response comments above) Changes to the MS: We do not intend to make changes to the MS in this regard. However, if advised by the editor, we may remove all stable isotope related aspects from the MS and change the title as follows: "High resolution  $^{14}\text{C}$  bomb-peak dating of tree rings of an African baobab from Oman"

Specific Comments/Suggestions I am going to focus here on just major topics that are in need of clarification to verify the fitness of the data shown. - p4, l111. It is stated that 10 trees were sampled by increment cores from four different orientations (NE, SE, SW, and NW). Do you mean four radii were collected per tree?! If yes, random tree rings were used for  $^{14}\text{C}$  analysis or just one tree and radii's? Please, clarify. Response: One tree, and one radii for  $^{14}\text{C}$  and stable isotope analysis Changes to the MS: We will be more specific the revised MS.

- p5, l148 to l55. How the tree specimen selected was dendrochronologically-secure? How the chronosequence of tree rings (prior  $^{14}\text{C}$  dating) was obtained without a master chronology for Baboab species?! The passage selected here describes just figure 2. Later (at p.6, l190 to 194), it is explained that no dated tree-ring width chronology from the study region is currently available. Response: One tree, and one radii. No chronology from several trees has been developed for this case study and no chronology from any other tree species from this site or region has been published. Changes to the MS: This will be added to the text.

- Therefore to anchor the chronosequence of tree rings (prior counting of all baobab tree rings) the  $F^{14}\text{C}$  of the TPBs and Oxcal was used instead. Is this correct?!...

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Response: No. One tree, and one radii was analysed. No chronology from several trees has been developed for this case study. Tree rings were counted and  $^{14}\text{C}$  data was used to validate the ring count. Changes to the MS: This will be added to the text. - If yes, this explanation should appear early on in the text. The fitness of the chronosequence is the backbone of the atmospheric  $^{14}\text{C}$  record production using tree rings. Response: One tree, and one radii. No chronology from several trees has been developed for this case study. Tree rings were counted and  $^{14}\text{C}$  data was used to validate the ring count. Changes to the MS: This will be added to the text.

- Plus, add what type of juniper species you are referring to. Response: *Juniperus seravschanica* Changes to the MS: This will be added to the text.

- p5&6, l159 to l65, and l177 to l178. Passages explaining the wood material used for radiocarbon and stable isotope analysis are confusing, and very troubling. It appears that the full dataset was produced in two phases, a pre-screening phase with 5 calendar years or so, where just 1/3 of the tree ring (cut parallel to the fiber orientation, in radial direction from the cambial zone) was used. In a second phase, in order to measure the remaining calendar years, just 2/3 of the single tree ring was used for  $^{14}\text{C}$  dating. The remaining material was then used for  $^{13}\text{C}$ . This description gives the idea that the tree ring cutting for isotopic analysis was selective, before chemical extractions took place. Response: As mentioned above, the tree rings of the baobab sample were counted. Since missing rings and miscounting was expected  $^{14}\text{C}$  bomb peak wiggle matching should help to validate the ring counting. In the first phase 5 individual tree rings covering individual, not consecutive years of the bomb peak period (as defined by ring counting) were selected for narrowing down the period. Changes to the MS: this will be exemplified in more detail in the revised text.

Normally a homogenized cellulose-extract of a full single whole-ring (from early- to late-wood) is used to reconstruct atmospheric  $^{14}\text{C}$  data. Response: Cellulose has been extracted for all samples of this study. Here, all tree-ring material, except from terminal parenchyma bands has been used for a  $^{14}\text{C}$  analysis. Changes to the MS: This will be

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added to the text.

It is understandable that since the baobab contains 69-88 % parenchyma cells, mostly concentrated at the terminal parenchyma bands (TPBs) or late-wood, this portion was removed. Response: Yes, parenchyma bands (TPBs) were separated from tree-ring material prior to  $^{14}\text{C}$  and stable isotope analysis. But, no further selective sampling was performed for  $^{14}\text{C}$  analysis. Tree rings of 1961-1963 and 2005 were sub-divided, but all sub-division were then analysed (see fig. 8) Changes to the MS: This will be clarified in the text and figs. 3 a,b will be modified to make this easier to comprehend.

But if the remaining material was further sub-divided by removing wood material representative of the growth season (Figure 2), unexpected  $^{14}\text{C}$  offsets would then be expected, especially at the slopes of the bomb peak. Accurate cutting of the tree rings is paramount for the reconstruction of atmospheric  $^{14}\text{C}$  data. This was already demonstrated by the intra-annual analyses of  $\text{F}^{14}\text{C}$  for the calendar years of 1962 and 1963 shown here. Moreover, if the wood cutting was indeed selective (prior chemical extractions, as mentioned above), Response: This concern is irrelevant. Tree rings were sub-divided for intra-annual stable isotope analysis and detection of pre- and post monsoon hurricanes. Except from separating the TPBs no sub-division of tree-rings has been made for  $^{14}\text{C}$  analysis, except for 1961-1963 and 2005. Changes to the MS: This will be clarified the text.

I do not understand how the ms can assert at the abstract that “considerable autocorrelation was found in the  $\delta^{13}\text{C}$  series, confirming incorporation of previous years’ carbon significantly affecting the average age of derived wood”, if the wood material tested was not the same. Analyses of  $\delta^{13}\text{C}$ ,  $\delta^{18}\text{O}$ , as well as  $^{14}\text{C}$  should be done from homogenized cellulose-extracts from the same wood aliquots. Please, clarify. Response: autocorrelation was found in min, max and mean of  $\delta^{13}\text{C}$  values of intra-ring sub-divisions. Tree-rings were sampled by dissecting sub-divisions of approx. 0.5mm. From each sub-division aliquots (=same mass (weight) from each sub-division) were taken, pooled together and homogenized for  $^{14}\text{C}$  analysis. The rest of each subdivi-

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sion was used for stable isotope analyses. Cellulose extraction has been performed on each individual sample for  $^{14}\text{C}$  and stable isotope analysis, respectively. Changes to MS: This will be clarified and exemplified in the revised MS.

-p5, 1164 & 165. Some of the TPBs removed were selected for  $^{14}\text{C}$  dating in phase one (4 or 5 samples). There is no mentioning of the chemical treatment they were subjected to prior sample processing for  $^{14}\text{C}$ -AMS. Please, explain. . . Response: Cellulose extraction has been performed on each individual sample for  $^{14}\text{C}$  and stable isotope analysis, respectively. It has been mentioned in the abstract and introduction that measurements were performed on cellulose. In paragraph 2.4 we outline that all “110 samples were holocellulose extracted with a base-acid-base-acid-bleaching procedure after (Němec et al., 2010). Ten samples were further purified to alpha-cellulose with an additional base treatment (17.5 % NaOH for 2 h at room temperature) followed by washing and freeze-drying.” Co-authors and project leaders Lukas Wacker and Gerhard Helle are well aware of the need for and importance of cellulose extraction and sample homogenization for stable isotope and  $^{14}\text{C}$  analyses. Various methodologies of cellulose extraction and sample homogenization are established in their laboratories at ETH, Zürich and GFZ, Potsdam since decades. The methods applied here are well adopted by the international scientific community and all methods used here were previously published in peer-reviewed scientific journals. We have indicated which specific procedure has been used and have referenced the corresponding papers (e.g. Wacker et al. 2010a, b; Nemeč et al 2010; Wieloch et al. 2011; Laumer et al. 2009 etc.) that describe these methods in detail. We initially thought that this might be sufficient and maintains the reading flow of the MS. Changes to MS: If given the opportunity by the editor we will be more specific and detailed in the revised MS.

-p.6, 1182 to 190. This portion is very confusing. The TPBs  $\text{F}^{14}\text{C}$  and OxCal were used to anchor the chronosequence. This would give a general idea of the calendar ages of these tree rings, which is ok. But since no chemical extraction appear to have being applied to TPB samples (no description of such is offered), Response: We disagree! As

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mentioned above, the MS unambiguously mentions that cellulose extraction has been performed and cites descriptions of the methods used. Cellulose extraction has been performed on each individual sample for  $^{14}\text{C}$  and stable isotope analysis, respectively. Hence, TPBs underwent cellulose extraction as well. Changes to MS: If given the opportunity by the editor we will be more specific and detailed in the revised MS.

I do not understand why one should expect that they would match with the NH<sub>3</sub>. Please, rephrase statements. Response: The study site is located in the NH<sub>3</sub> zone as outlined by Hua et al. 2013. Hence, one could expect or hypothesize that all plant material containing carbon photosynthesized at this site should have the isotopic signature ( $^{14}\text{C}$  and  $^{13}\text{C}$ ) of atmospheric CO<sub>2</sub> prevailing at this site. Changes to MS: We will attempt to be more specific and clearer in the revised MS.

Regarding figure 3, and text portion between 187 to 190. What do you mean w/ “the baobab samples’ position on the time axis is relative to their position within the tree ring of a growing season lasting from June until September”? Were the calendar years in the “x-axis” of figures 3 (and figure 8, as well) adjusted to match w/ the growing season of the baobab species as shown in Fig. 1C (June to September)? It is hard to see if such adjustment was applied in figure 3, as the figure is small. But I think that this adjustment was not applied to figure 8, as it should, and therefore the entire baobab F<sup>14</sup>C values are too far to the left. Have you take this monthly shift in account in the modelling as well? This calendar year adjustment should also appear at Table 2, second column to avoid confusing between growth date and dendro-date. Response: We will more clearly write how we positioned our samples on the x-axis. We think however, that the positioning was properly done, but maybe not easy to read in the graph. That is why we did a zoom-in on figure 3 (where you can better see the positioning in the relevant part). Nevertheless, we will check everything once again.

On figure 3A, I am left unsure (without checking all records in Hua et al. 2013 supplementary material) the main differences in uncertainties between SH3, SH1-2 and NH3 records beyond about 1972 (orange shaded area). Why is this shaded area particularly

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different from all others, when the SH3 record (based on Muna Island data) stopped in 1979? Beyond this calendar year most records assume no differences between hemispheres due to scarcity of data in the tropics. Please, explain. Response: We disagree! We are convinced that the data set published by Hua et al. 2013 is correct and was cited by us correctly. We kindly ask for your understanding that we cannot recap in all detail the paper by Hua et al. 2013. Please do note that SH3 is not different from all others, it just carries larger uncertainties, likely due to the fact, that Muna Island stops in 1973.

Figure 3B, I appreciate the effort of showing F<sup>14</sup>C values between the calendar year of 1962 to 1964, but further discussions on air mass circulation (as mentioned earlier) are still lacking. Since the citation of Nydal & Lovseth 1983 is already listed in the article, all other records in the same zonal band in this article should be added to the plot. Response: Thank you for this comment. Changes to MS: We will add a discussion on potential effects due to atmospheric circulation and also add the data to fig. 3b in the revised MS.

Second, most of the citations in this figure legend are not in the reference list. Third, replace Turnbull et al. 2017 by Turnbull et al. 2017. Response: Thank you for this comment. Changes to MS: We will correct the references and the reference list in the revised MS.

-p.9, section 3.1. I do not understand why one should expect that the TPBs would match with the NH<sub>3</sub>, or even match with the TRs (holocellulose extracts, Table 2). I don't see how this part is relevant. Response: As mentioned above: our study site is located in the NH<sub>3</sub> zone as outlined by Hua et al. 2013. Hence, one could expect or hypothesize that all plant material containing carbon photosynthesized at this site should have the isotopic signature ( $^{14}\text{C}$  and  $^{13}\text{C}$ ) of atmospheric CO<sub>2</sub> prevailing at this site. Since our data show a clear mismatch we think this is important to mention. In other words, how can one expect plant material (like wood or specific wood cells like TPBs) to not show the atmospheric carbon signature of the region it was growing?

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Changes to MS: We suggest no changes to the MS in this regard.

Most importantly would be comparisons between F14C data of TRs and alternative alpha-cellulose treatments that target the removal of starches and sugars (e.g., “Soxhlet”-type extractions using solvents). Note that the alpha-cellulose extraction described here was attained by adding an extra step of 17.5% NaOH to the holocellulose procedure. Incomplete removal of resinous compounds during chemical pretreatment of tree rings biasing 14C data has been shown by others (Cain and Suess 1976, Westbrook et al. 2006, for example). Response: As mentioned above: Cellulose extraction has been performed according to well established methods (cf. citations in MS: Wacker et al. 2010a, b; Nemeč et al 2010; Wieloch et al. 2011; Laumer et al. 2009 etc.). Please do note that angiosperm baobab wood does hardly contain resinous compounds and if so resins, as well as other extractives (e.g. starch, sugars etc.) would have been completely removed by the procedures applied (for review of established methods, including methods applied in this MS please cf. Helle, G., Pauly, M., Heinrich, I., Schollaen, K. (2020). Stable isotope signatures of wood, its constituents and methods of cellulose extraction. In: Siegwolf, R., Brooks, J.R., Roden, J., Saurer, M. (eds.). Springer: Tree Physiology Book Series.) Changes to MS: We will try to rephrase our methods section in order to be clearer and more specific.

-p12, section 4.1. I found this section highly speculative; especially when no 14C dating targeting starch extracts from the baobab parenchyma-dominated wood was attempted. Response: Solvent extractives (starch sugars, etc.) were all removed. Changes to MS: We will be more specific in the revised MS.

Richardson et al. (2013), cited in this section, indeed found direct evidence for ‘fast’ and ‘slow’ cycling reserves in stemwood. However, Richardson et al. (2013) also stated that even though aboveground temperate forest trees contained very old pools of starch and sugars, stressed trees would still use up first all available present-day fast cycling carbon pool to support growth and metabolism. This would include even the most recently added starch molecules. Therefore, the usage of “older” NSC reserves

C13

was set for times of stress. Richardson et al. (2013) did not mention that ring cellulose 14C results were inaccurate after direct comparison with the northern. Response: We disagree that “older” NSC reserves are used for times of stress only. Deciduous trees like baobabs do need carbohydrate reserves for bud growth, leaf emergence and maintenance metabolism during the dormant period. That is why we have assumed a contribution of “old carbon” of 15%. Please do note, with the simple model considerations presented here we do not intend to precisely describe or capture temporal changes of carbohydrate pools in baobab trees. We can show, however, that the F14C values of baobab tree-ring cellulose can better be explained when assuming a mixing of pools of carbohydrate precursors of cellulose. Changes to MS: We will be clearer and give more details on this aspect in the revised MS.

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