

Review of “The triple oxygen isotope composition of phytoliths as a proxy of continental atmospheric humidity: insights from climate chamber and climate transect calibrations” by Alexandre A. et al. BGD.

**General:**

The manuscript deals with the potential of using phytoliths, micrometric amorphous silica particles that form in continuously living plants, for paleoclimate reconstruction of relative humidity changes. These humidity changes are difficult to reconstruct but are highly important to understand the drivers of climate changes on Earth. Studies on phytoliths have been conducted since long in various research fields such as archaeology, biology, plant physiology as well as paleoclimatology. With the significant improvements of online measurement systems – that are much easier to handle – the amorphous silicate analysis have strongly increased. This includes also the isotope determination of the oxygen that is directly attached to the silica, hence the non-exchangeable oxygen. There are several difficulties to circumvent to come up with reliable and meaningful results. One of the most important issues is to remove the exchangeable OH groups of the silica. Recent developments seem to completely overcome this shortcoming and opens up new opportunities for research using stable isotopes, in particular, in view of the newly available measurement techniques for stable oxygen isotopes, including  $^{17}\text{O}$ . Therefore, the presented work is timely by combining laboratory work and field analysis using state-of-the-art measurement techniques. The question addressed with their research, namely to investigate the relation of the  $\Delta^{17}\text{O}$  to the relative humidity is important since there is hardly any known parameter available that could be used for paleoclimate work. The authors are experienced to work on phytoliths and/or using isotope analysis for their studies. This is again a well taken combination which I very much appreciate.

I suggest accepting this manuscript after addressing the following points:

**Major points:**

- Since there have been different measurement techniques used to determine the oxygen isotopes, it would be worthwhile in this context to report the comparability of the results mentioned in an additional table ( $\text{H}_2\text{O}$  on Picarro L2140i and  $\text{O}_2$  converted from  $\text{H}_2\text{O}$  on Delta V mass spectrometer) as well as the measurements done on the Picarro micro combustion module (MCM) in comparison with direct water measurements.
- How was the difference in  $\Delta^{17}\text{O}$  between Phyto and LW calculated since from Fig. 1a, I am not able to obtain Fig. 1c for this difference? Please check it. This is also in line with the slopes of LW and Phyto vs. RH being different.
- The comparison of the field with the lab results are critical (line 410 to 417), since there is no reason given why we should take the RH only for those months with a limited precipitation. This is in particular important since the  $r^2$  values actually decreases when going from RH or RH15 to the range limited by precipitation. This requires further discussion. It is no argument to fit the field data to the lab data just based on a slope measured.
- A weak point is indeed that no water vapor measurements are performed, this is indeed a strong shortcoming because a Picarro L2140i was available for the study. Yet, the authors clearly pointed out the importance to include such measurements in future studies. Was the leaf water measured for  $\delta\text{D}$ ? If yes, this may help you with the interpretation in that it helps to make reasonable assumptions for the water vapor values.

- Triple isotope comparison of Phyto with RH: It would be nice to distinguish the LW values given in blue for the high RH values (80-100 %) compared to low RH values (40%). This would allow the reader to better follow fig. 6. You may also use ellipses for these clarifications. Same issue with the Phyto values given in red.

#### Minor points:

- Why do you clean it cryogenically for NF, is NF produced during the fluorination process? How much could it affect the  $^{17}\text{O}$  and therefore the  $\Delta^{17}\text{O}$  results?
- You mentioned that you checked the temperature independencies for  $^{18}\text{O}$  and  $^{17}\text{O}$  up to 70°C. Please add more information on this issue, because this is important. How have you done it? Wouldn't it be worthwhile to show the experimental results that you have obtained in this paper?
- There is a significant difference of one of the standard material used, i.e. San Carlos Olivine. Whereas Sharp et al. (2016) reported a normalized  $\delta^{18}\text{O}$  value of 5.3 ‰ and a 17O-excess value of -54 per meg your values were  $\delta^{18}\text{O SC} = 4.949 \pm 0.219$  ‰ and 17O-excessSC =  $-49 \pm 24$  per meg. Why this difference in  $\delta^{18}\text{O}$ ?  $\delta^{18}\text{O}$
- On line 317 you have used ppm to express  $\Delta^{17}\text{O}$  whereas you have often used per meg, be consistent over the whole manuscript.

#### Specific remarks:

I. 289: were dehydrated... do you mean adsorbed water or interstitial water?

I. 345: Make sure the minus sign is attached to the number.

I. 362 etc.: Make sure that only relevant digits are given for the measurements according to their uncertainty.

I. 366: I suggest changing....withdrawn from the data set... to ....excluded from further calculations...

I. 388: delete 00 prior to the number 2.

I. 538: add space after for

I. 542f: One can expect **that** the isotope composition...

Table 1: Explain P1-40-29-04-16 etc. in the table legend.

Table 2: Legend not consistent with table.

Fig. 1: add x-axis on the top as well for easier readability. Panel c) is not consistent for me since it should be the difference between the measurements shown under panel a). This is not correct for all points. There should be an increase in Phyto-LW. Am I wrong?

Fig. 5: How relevant is this figure?

Fig. 6: explain the different slope and slope ratios used in the figure.