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Interactive comment on "Combination of soil water extraction methods quantifies isotopic mixing of waters held at separate tensions in soil" *by* William H. Bowers et al.

Anonymous Referee #2

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This paper presents some progress on the centrifuge technique to separate soil water held at different bindings strengths into the potential stable isotopic pools that may exist in soils. I think this study has some good contributions to offer, but I also think it needs some improvement before I can endorse its publication in HESS.

General comments:

I found the analysis and discussion of the results to be quite "thin". By that, I mean that there is not an especially in depth or nuanced explanation and discussion of many components throughout. Specific examples follow, but in general, I suggest that the senior authors of the manuscript return to it with a more discriminating eye and identify

where it can be "deepened".

The authors base the rational for conducting the study on making progress on identifying the potential soil water reservoirs (isotopic or otherwise) that underpin the ecohydrologic separation, or "Two Water Worlds" (TWW) hypothesis. However, there is only the most minor discussion of this concept in the introduction, and then the authors return to it throughout the results and discussion citing how their findings apply to TWW. This is problematic because the reader doesn't have any firm understating of TWW or how the authors are interpreting TWW (because interps vary). I suggest there be a fuller discussion of TWW and how this study specifically contributes to investigating it in the introduction.

Because HESS has an open review process, subsequent reviewers have the advantage of seeing previous reviewer's comments. That is the case here, and while I do not intend to "pile on" the authors, I do support Reviewer #1's comments, especially in regards to the mixing analysis (see my specific comments below).

Specific Comments:

L44: Need a brief explanation of what in situ equilibration is and some references of papers using either of these methods.

L100: These waters aren't all that different in isotope compositions. Nota Bene: Kona Deep drinking water is about 0 ‰ in δ18O and δ2H and is available on Amazon.

L107: The abstract claims that the light water was enough volume to fill only the smallest pores. The procedure described here seems very arbitrary.

How do you have any confidence or measure of what soil pores where filled and to what extent?

L118: Are you sure this was the extraction temp? Did you use boiling water? Laramie is pretty high elevation and thus water has a low boiling point.

L122: 95% is still not ALL of the water.

L127: You use the Two Water Worlds terminology here, but you haven't ever really discussed t in any detail in the introduction. I suggest you do so, to help contextualize the rest of the paper.

L136: Three and four hours seems like a long time! On what basis did you choose these times?

L138: You never really discuss what is tightly or highly bound, or what the potential mechanisms for this soil water are. There are many aspects to this, from soil pore size, to soil mineralogy, etc. This is a main concept of your paper, but you never give the reader any background or basis of understanding how you are using this terminology and "boundness" concepts.

L140: Again, are you sure it was ALL of the water left. Or was it 95%? I dont mean to be tedious here, and there are limits to CVD, but that is prwcisely my point. Even CVD at 100 C wont get all the water out that is in interlayers spaces in clays, etc. Are more nuanced discussion is needed (maybe it comes later in the discussion), and at least some acknowledgement of the study's potential limitations is needed. I will look for that as I read. . .

L142: I think you should move up the details about the centrifuge and inserts. It hard to envision what you did until you tell us about the inserts.

L149: Good that you accounted for evap during the procedure. I assume it was done at room temp, but I could easily see temp being higher inside the centrifuge, especially for 3 to 4 hours. Did you measure this?

L167: What is atomic fraction? Do you mean isotope ratios (not in delta format)? Or do you mean mixing fraction?

L178: Is this 1% the total mass (water + soil) or just water? If it was total mass, then a decent amount of water lost to evap (and a big shift in isotope ratios) could be contained

in the 1% number. I suggest a sensitivity analysis be done to quantify (in isotope terms) what the effects of this much water loss would actually be. It may seem tedious and unnecessary, but with this much handllng of the wet soil, I could easily see evaporation being a bigger factor in isotope results than a casual view would expect.

L217: You state that the BSE waters were not significantly different from the applied waters, but in Figure 3 upper left panel they sure look different to me. It seems that you were not getting back what you put in. This seems problematic.

L218 / Figure 3: Suggest adding A -F labels to the panels in Figure 3. Also, the x-axis labels and ticks seem inadequate.

L222: I suggest keeping the applied water points in all panels in Figure 3 for easier comparison. Perhaps make them dashed outline or ghosted or something to show them but not distract form the time series data.

L227: So basically, after enough time, all the waters extracted by any means all converged upon the Heavy water signature. And the heavy water signature is the one that you soaked the sample in, but only put a little of the light water in the same samples?

L231: So, the conclusion is that the samples were all well mixed? OR something else? Because the dont look well mixed to me, especially not until dy 3 or later. Am I missing the point? If so, please explain better.

L243: How do you evaluate the mixing results if you dont actually known how much of each type of water you put into the soil? Seems to me that with so much more heavy water than light, you are not really evaluating mixing, but more like the time to equilibrium, wherein the heavy water signal just overwhelmed the light because there was so much more of it.

L248: This section reads more like a conclusions paragraph than the start of a discussion. You haven't really supported any of these statements, yet.

L252: What are the proposed mechanisms of mixing? This is hard to determine, because you haven't ever discussed where in soil water is actually held. Is the "mixing" done via diffusion? if so, water self-diffusion in soils is fairly well studied and you could greatly increase the impact of your findings by bringing in some discussion of that work. This seems like an over simplistic analysis of your results, which are a bit fast and loose as it is. No offense intended, just that I am seeking more detail and justification in your measurements and results.

L266: Are there carbonates in your soil? Easy test with HCl.

L270: This is the first time you have acknowledged that your samples have perturbed soil structure and thus pore sizes. This may be the biggest reason for any isotope effect of any discussed.

L277: Finally, the discussion I was yearning for. Can you expand by making some calculations that support these arm waving statements?

L288: Good point on the pore size changing during the spinning.

L290: Yes, shorter spin times!

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