This study examines mechanisms of soil organic matter (SOM) persistence. The manuscript is well written, the experimental design is sound (though limited to a single soil), and the conclusions drawn are consistent with the data collection. The study is particularly compelling because it pulls together two older or more conventional methods for studying SOM persistence (fractionation and radiocarbon) with newer energetic approaches using ramped combustion thermal analysis. The authors have demonstrated where the data from these various sources clearly overlap and can be interpreted consistent, and more importantly, where the data appear to be inconsistent or in conflict. The conclusion that I reached in reading the manuscript is that physical and chemical fractionations to not isolate pools of SOM that are consistent with our conceptual representation of them. I’m not sure the authors would agree, but if so, this point might be made more clearly and succinctly. Overall, I think the manuscript is worthy of publication, but have several specific comments and suggestions for revision outlined below.

Abstract

Ln22-25: These two sentences and the following one seem to be in the wrong order. The authors might want to rethink the logical sequence of ideas they are trying to convey.

Ln24: Would “is better” a more precise wording than “could be” for what the authors are trying to assert? They seem reluctant to be prescriptive, but I think that reluctance reduces the impact of the study.
Introduction

Ln32: Adverbs like “remarkably” are too subjective and don’t do any narrative work. It can be removed or elaborated to describe what is remarkable if the authors feel it is important. This is true throughout the manuscript.

Ln33: Can the author be more precise than “varying” persistence? For instance, might a range of ages/MRTs be provided to demonstrate this variance?

Ln39: I think this gets adequately elaborated later in the paragraph, and I’m also not well versed in the nuances of 14C analyses, but because soils are open systems I thought we were supposed to avoid terms like “age” and instead invoke residence/transit time. Perhaps provide more precise diction here?

Throughout the manuscript, the authors suffer a degree of “recent-ism” when it comes to citations about physical and chemical fractionation. While the work by Cotrufo and colleagues is excellent, some might not consider it seminal because it reframes and revisits much of the work that was done ~10 years prior. For instance, Gregorich et al. (2006) is among a number of excellent papers that clearly conceptualizes and defines “uncomplexed” organic matter and elaborates on physical fractionation. Similarly, there is excellent work published by EA Paul and SE Trumbore on 14C work on chemical fractionation residues that is uncited in favor of more recent work. While citation of recent work is not a problem in itself, it can be problematic when presented as seminal when it may not be.

Methods

Ln160: I believe figures are to be numbered in order of mention in the text. The author might consider referring to Figs S1 and S2 here, or renumbering. Also see my comments below about Results section 3.1.

Ln164-175: I recommend moving this paragraph to ln196-197. This paragraph is about data and it interrupts the narrative about the instrumental and analytical methodology. The narrative might instead be about the collection of observational data, followed by the manipulation and analysis of this data.

Ln166: Values for the fitting parameter lamba are important in determining the shape of the energy distribution curve. These values should perhaps be reported for reproducibility.
The notation around “E” can be a bit confusing. There is E, μE, σE and Ea. Be sure that the nomenclature is well defined at the outset and consistent through the manuscript.

Results

Unfortunately, this assertion is untrue. I quick search of “soliTOC” will reveal a few recent papers using the instrument for thermal fractionation of SOM. Perhaps the authors need to be more precise if they mean the coupling with 14C analyses.

Section 3.1: While the QA/QC of the method development is very important, I would strongly recommend moving it to the supplemental materials. Reproducibility and accuracy is essential and no other part of the study would be valid without it, but I find that it doesn’t adequately contribute to the core narrative developed in the discussion and conclusion to warrant leaving it in the main body.

Table 1: Units in the headers should be “J”, not “j”. Also, should “E” not be “μE”?

Table 2: Units in the headers should be “J”, not “j”. Also, should “E” not be “μE”?

It’s not clear to me why third level numbering is needed if there is only one item in the list. Perhaps the structure and numbering scheme can be revisited.

The authors invoke a p-value without any prior description of the approaches/methods for statistical analyses.

Figure 1: Axis title for the dependent axis is missing. While it is clear from the caption what it should be, the title should be in the figure. Also, the unit for the independent axis should be “°C”, not “C”.

Figures 3 and 4: Perhaps the axis titles could be elaborated/defined for clarity? Eg, “14C Fraction Modern (Fm)” and “Activation Energy (μE)”. Also, replace the letters with greek letters/symbols in the axis titles.

I believe the authors mean “temperature” instead of “temperate”.

Discussion
Overall, I found the discussion to be compelling but a little overelaborated or structured. The complex observations and data do result in a complex set of interpretations, but I think the reader would benefit from a shorter, clearer and more succinct discussion. Here is the message I got when reading the discussion as a possible guide for improving clarity. Placed in our current conceptual framework, physical and chemical fractionations generate pools of SOM with predictable/anticipated mean ages, but not anticipated distributions of ages or “persistence” as expressed by thermally derived activation energies. This calls into question either fractionation or thermal analysis as a means of assessing SOM stabilization mechanisms and persistence. Observations seem to suggest that density or chemical dissolution/oxidation don’t generate fractions consistent with our predictions based on conceptual framework. This calls to mind Smith et al (2002) on measured fraction vs. model pool. It seems fractionation may not successfully separate SOM into the distinct pools we hope for. In the end, thermal fractionation can successfully elucidate age distributions of SOM, but works best when free POM is removed.

Ln505: I’m not sure I agree. Rather than integrating stabilization mechanisms, some fractionation methods are designed to isolate pools of SOM based on specific mechanisms (eg, mineral-associated vs. uncomplexed; easily oxidized vs. recalcitrant). Perhaps this paragraph needs to be rephrased or rethought.

Conclusions

I guess the sense that the authors are restraining themselves from being too prescriptive and are writing prudently. I would encourage providing the reader with a clearer (if not stronger) message based on the outcomes of the study. Concisely but specifically, where do the authors think the problems/shortcomings/fallacies lie, and what can be done to avoid them?