

## ***Interactive comment on “Particles under stress: Ultrasonication causes size and recovery rate artifacts with soil derived POM, but not with microplastics” by Frederick Büks et al.***

**Frederick Büks et al.**

frederick.bueks@tu-berlin.de

Received and published: 23 October 2020

Dear referee #2.

Many thanks for your proofreading and your helpful comments. We added the requested data to our manuscript and supplements. In the following, we want to explain how we propose to improve our explanation as in your favor.

Comment 1: For oPOM and pyrochar, the recovery rate decreased with the increment of ultrasonication power. The cause was supposed to be an increase of new active surface to absorb the comminuted oPOM after disintegration of soil aggregates. However,

C1

this explanation is unlikely applicable to pyrochar.

[1] We hope that we understood your comment correctly. Pyrochar has an enormous internal surface, but we assume that also pyrochar receive a larger outer surface if particles are comminuted. However, based on the comment of referee #1 on the lack of evidence in literature regarding the association of small organic particles with sand grains we refrain from this explanation and instead added reply [22] to referee #1.

Comment 2: As mentioned in Ince (2001) and confirmed in Kaiser & Berhe (2014), ultrasonication induced high temperature may reduce total C content due to oxidative reactions. If this happens, the conclusion of “counting up to around 36.2 to 64.2 % of POM to the MOM” is really overestimated. I would like to know how much oPOM was lost and how much was transferred to MOM in this study.

That is a very interesting question, which is really improving our work. We did measurements in this regard and added after line 164 into the material & methods section: "2.5 organic matter balance: A second set of triplicates of pyrochar and farm soil oPOM were treated similarly at 0 and 500 J/ml to balance the complement of the recovered POM. For this purpose, the C concentration within the lyophilized sediment was measured by use of a CNS analyzer and converted to POM mass by use of the C content (%) of the respective organic matter. In addition, the mass gain of the cellulose acetate filters was measured after rinsing the sample and drying the filter at 70°C for 24 hours. The DOC concentration of the filtrate was measured and converted to DOM by use of an assumed 50% C content. The difference of these and the recovered fractions compared to the initial weight of organic particles is termed the balance loss during the extraction procedure."

Corresponding to that, we added the following to the results section after Line 179: "3.3 Mass loss: The treatment of pyrochar triplicates with 500 J/ml resulted in a recovery rate of  $54.3 \pm 5.2$  % after density fractionation. In turn,  $34.9 \pm 3.7$  % of the POM remained in the sediment,  $0.6 \pm 0.1$  % into the DOM fraction and  $<0.5$  % onto the filter,

C2

leading to a balance loss of  $10.2 \pm 2.1$  % (Fig. 2). The respective data of farm oPOM are  $54.6 \pm 1.9$  %,  $20.3 \pm 3.1$  %,  $5.1 \pm 0.2$  %,  $<0.5$  % and  $20.0 \pm 1.5$  %. Samples treated with 0 J/ml instead showed a significantly higher recovery rate and lower retention compared to the 500 J/ml samples. In contrast, the balance loss remained constant between 0 and 500 J/ml." The data are shown in an additional figure and added to the supplements.

We furthermore supplemented our comment [22] to referee #1 in the discussion section as follows: "We assume a mechanism that prevents POM from detection. This effect appeared in our experiment from energies around 50 J ml<sup>-1</sup> with the beginning destruction of oPOM. As mentioned in Ince et al. (2001) and confirmed in Kaiser and Berhe (2014), ultrasonication induced high temperature may reduce total C content due to oxidative reactions, but the balance loss, constant between 0 and 500 J/ml in both pyrochar and farm oPOM, implies that there is no burning of organic matter due to ultrasound treatment. The recovery rate decreases in the same degree as the retention in the sediment increases when ultrasound is applied, while relics on the filter and lost DOM, which doubled on a low level, play a minor role. Extreme thermal conditions occurring during ultrasoincation, however, may explain the retention of POM within the sediment increasing with higher energy levels. Sparse data on molecular alteration ..."

Comment 3: "About 100 mg POM were suspended" for particle size analysis. However, the initial quantity of oPOM in each vessel is  $20 \text{ g} \cdot 0.5\% = 100 \text{ mg}$ . Therefore, with a recovery rate may be as low as 50%, it is unlikely to get 100 mg of oPOM for particle size analysis.

[5] We are sorry for this phrase has escaped our notice. It actually means "up to 100 mg" and refers to the plastic samples, which had an initial weight of 0.2 g and were recovered by nearly 100%. For pyrochar and the oPOMs the QicPic used a smaller amount according to the extracted matter. The actual range of sample weight is therefore "30 to 100 mg", which is to correct in line 149.

C3

Comment 4: The farm and forest soils used for this experiment were from an organic horticulture and a spruce/beechn mixed forest. However, soil organic C content was only 4.9 and 7.3 g kg<sup>-1</sup>. Please check these data.

[6] Thank you for your mindful reading. It is indeed 4.93% and 7.32% (or 49.3 mg/kg and 73.2 mg/kg) and will be corrected in Lines 100 and 102.

Comment 5: Is the weight of POM measured or the C content measured?

The recovery rates base on POM weights. That is because (1) this work focus on mass losses and (2) C analytic is destructive and would have doubled the operational effort with respect to the following particle sizing.

Comment 6: There are some grammar errors, including explanation of the calculation of CF.

We thoroughly reread our manuscript and corrected some grammatical errors that had escaped our notice.

Best regards,

Dr. Frederick Büks, M.Sc. Gilles Kayser, M.Sc. Antonia Zieger, Prof. Dr. Friederike Lang and Prof. Dr. Martin Kaupenjohann

---

Interactive comment on Biogeosciences Discuss., <https://doi.org/10.5194/bg-2020-204>, 2020.

C4