



# Sequential Extractions for Analysis of Carbon and Nitrogen in Bulk Soil and Mineral-Associated Organic Matter

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## BACKGROUND

- Plants need nitrogen (N), but exactly how, when, and from where they access soil N remains unknown. Recent work shows that mineral-associated organic matter (MAOM) is a critical but overlooked pool of soil N.<sup>1</sup>
- We seek to understand what MAOM-N is comprised of, how it is distributed across and associated with different soil mineral pools, and how it differs from bulk soil N. Sequential dissolution extractions are commonly used to quantify MAOM-carbon (C) pools<sup>2</sup>, but have not been optimized for investigation of MAOM-N.

## HYPOTHESES

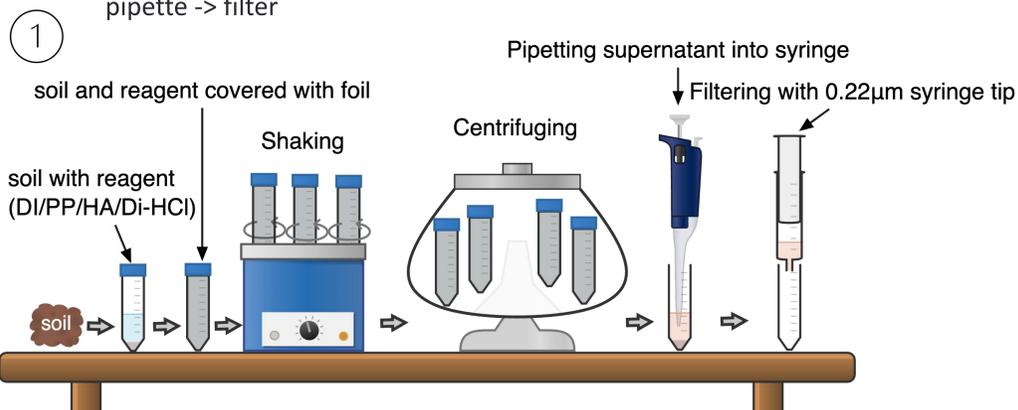
- MAOM and bulk soil N and C concentrations vary across soil types.
- MAOM N and C concentrations are greater than those of bulk soil.

## OBJECTIVES

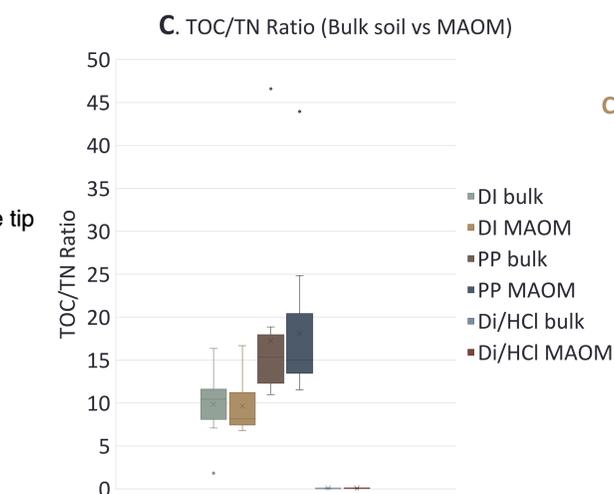
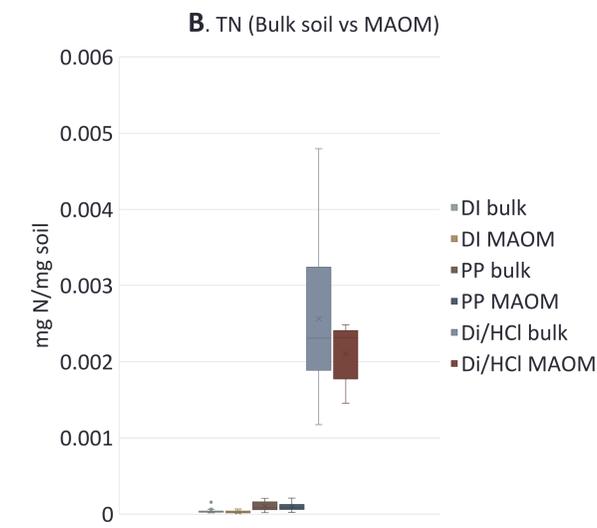
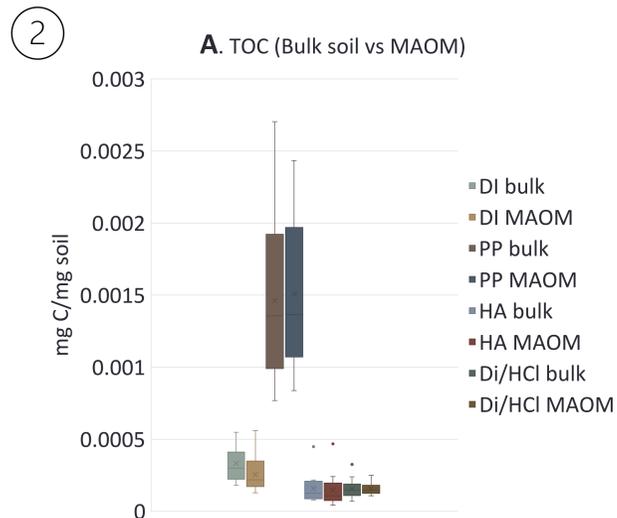
- Optimize sequential extractions to measure MAOM-N; Assess whether dithionite-HCl extract contains trace N from hydroxylamine.
- Compare N and C pools present in soils with different mineralogy and land use history.
- Compare N and C pools present in bulk soil and MAOM.

## METHODS

- We used 4 extractions in this experiment:
  - DI water extraction (DI):** dissolved organic C, soluble salts, water-soluble amorphous minerals
  - Pyrophosphate extraction (PP):** complexed Fe, Al, organic C
  - Hydroxylamine extraction (HA):** for non-crystalline inorganic Fe, Al, and organic C; slight dissolution of silicate minerals
  - Dithionite-HCl extraction (Di/HCl):** crystalline Fe-oxy(hydro)xides (hematite, goethite, lepidocrocite, and ferrihydrite), but not silicate minerals.
- Figure 1.** Extraction process: reagent -> foil -> shake -> centrifuge -> pipette -> filter



## RESULTS



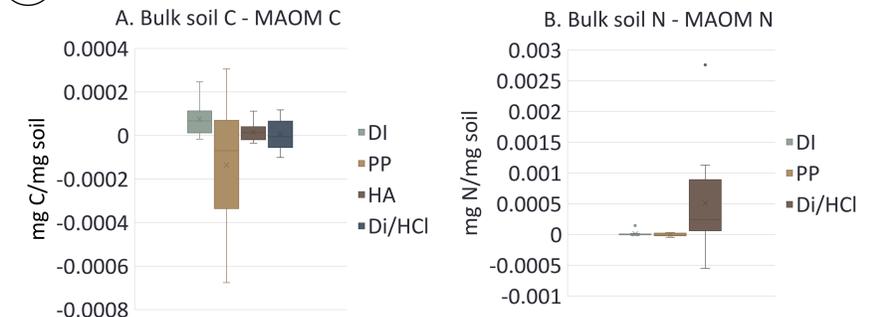
**Figure 2. TOC, TN, and C/N ratios measured with each extraction from bulk soil and MAOM.** Figs 2b & 2c only show 3 extractions because the HA (H<sub>2</sub>NO, hydroxylamine) contains N that obscures a signal from the sample itself.

**A. Total organic carbon (TOC) concentrations.** The PP extraction contains the highest TOC concentrations. Within each extraction, the bulk soil and MAOM contain similar levels of TOC.

**B. Total N (TN) concentrations.** The Di/HCl extraction contains the most TN (organic and inorganic N). This is because the order of extractions is DI -> PP -> HA -> Di/HCl. Although we did our best to remove the residual HA prior to the Di/HCl extraction, the amounts remaining still affected the N measured. Within each extraction, the bulk soil and MAOM contain similar N concentrations.

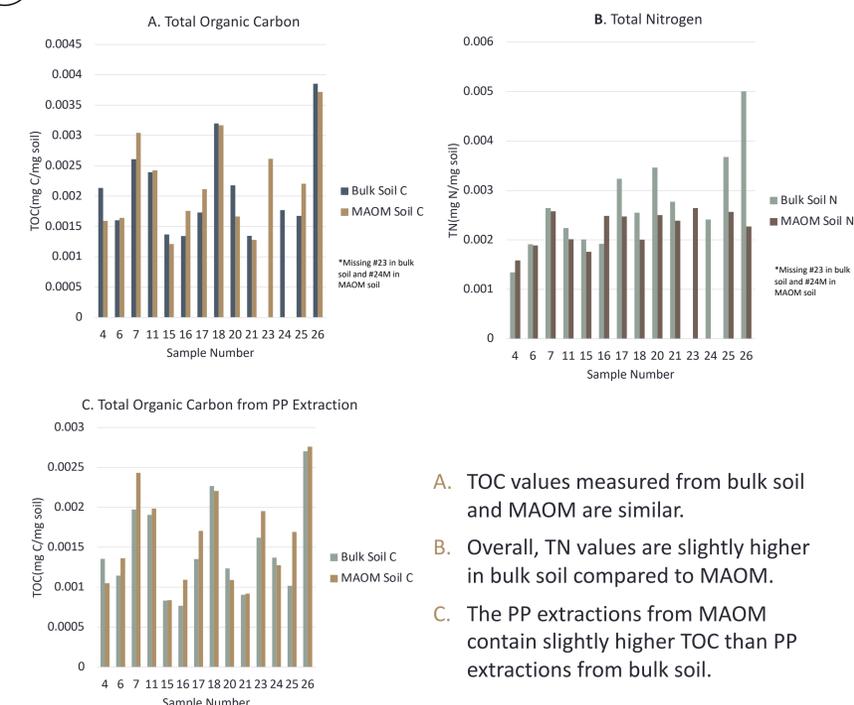
**C. TOC/TN ratio.** The PP extraction contains the highest TOC/TN ratio. The low TOC/TN ratio in the Di/HCl extraction is likely due to residual N from HA. Within each extraction, the bulk soil and MAOM contain similar TOC/TN ratios. We anticipate that the TOC/TN ratios will be larger because they will not include inorganic N.

3 **Figure 3.** Difference between bulk soil and MAOM from each extraction



- There is a wider range of differences in TOC measured from bulk soil vs MAOM with the PP extraction; median values are similar across extractions.
- There is a wider range of differences in TN measured from bulk soil vs MAOM with the Di/HCl extraction; values measured with DI and PP are similar.

4 **Figure 4.** Comparison between bulk soil and MAOM from each sample



- TOC values measured from bulk soil and MAOM are similar.
- Overall, TN values are slightly higher in bulk soil compared to MAOM.
- The PP extractions from MAOM contain slightly higher TOC than PP extractions from bulk soil.

## CONCLUSIONS

- MAOM TOC and TN contents vary across soil types, but are not very different from bulk soil TOC and TN. Further analysis is necessary to obtain organic N data for accurate comparison of TOC and TN. Future work will include two sets of extractions: one with all 4 extractants for mineral quantification and the other without hydroxylamine for N quantification under N-free conditions.

## REFERENCES

- Jilling, A., Keiluweit, M., Contosta, A.R. *et al.* (2018) Minerals in the rhizosphere: overlooked mediators of soil nitrogen availability to plants and microbes. *Biogeochemistry* 139, 103–122.
- Heckman, K., Lawrence, C.R., Hardin, J.W. (2018) A sequential selective dissolution method to quantify storage and stability of organic carbon associated with Al and Fe hydroxide phases. *Geoderma* 312, 24–35.