# The role of wetting vs. drying

on SOM destabilization

Kaizad F. Patel

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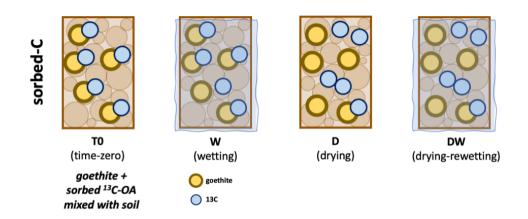
# **Objectives**

To partition biochemical and physicochemical protection/destabilization mechanisms for soil C by their sensitivities to wetting and drying.

# Hypotheses

1. drying will have a stronger effect on soil C destabilization, compared to wetting

# Experiment

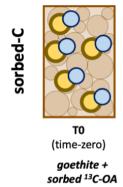


13C-labelled organic substrate was adsorbed onto clay and added to soil

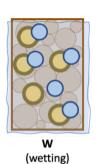
#### **SOIL USED**

- Palouse soil
  - Fine-silty, mixed, superactive, mesic Pachic Ultic Haploxerolls
  - 2.61 % w/w gravimetric moisture
  - 3.17 % carbon
  - 13C at% 1.08 (R = 1.09 %, δ13C = -26.9 %)
- the soils were sieved through 4 mm and homogenized
- stored at 4 °C until ready
- 60 g field-moist soil (~58 g oven-dry equivalent) was weighed into pint size Mason jars
- 5 mL deionized milli-Q water was added to each jar, and then the soils were held at 21 °C for 24 hours before the experiment began (conditioning)

### Experiment

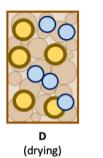


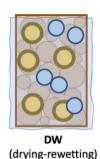
mixed with soil



goethite

**13C** 

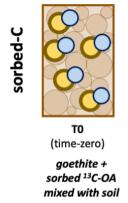


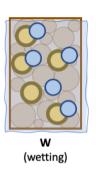


#### **PREPARING SUBSTRATE + CLAY**

- labelled substrate (13C-oxalic acid) was added to the soil, adsorbed onto goethite
  - labelled and unlabelled OA were mixed to prepare a solution of 150 mgC/L with at% = 16.02 (R = 19.08 %, δ13C = 15,975 ‰)
  - 20 mL of this solution was mixed with 5 g goethite
  - the goethite was rinsed with DI water 2x
  - 5 mL DI water was added to the rinsed goethite to form a paste
  - this paste of goethite + OA was added to each Mason jar
  - the final prepared goethite contained 0.25 % carbon with at% 3.94 (R = 4.10 %, δ13C = 2644.38 %)

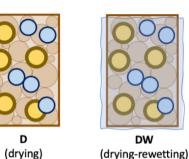
### **Experiment - Treatments**





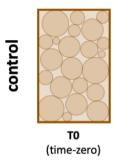
goethite

**13C** 



- The soil was subjected to one of the following treatments, after which the jars were sealed:
  - 1. time-zero: jars sealed as soon as goethite was added
  - 2. wetting: 20 mL water added
  - 3. drying: soil allowed to air-dry until constant weight
  - 4. drying-rewetting: soil allowed to dry, then rewet with 20 mL water
- The jars were sealed for 48 hours.

# **Experiment - Controls**



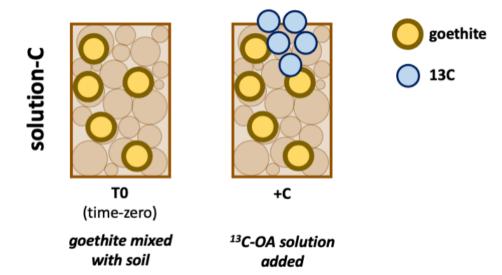




#### **CONTROL**

- A set of controls was analyzed by subjecting soils to the same treatments, but without the added goethite + OA
  - time-zero: jars sealed as soon as goethite was added
  - wetting: 20 mL water added
  - drying-rewetting: soil allowed to dry, then rewet with 20 mL water
- The jars were sealed for 48 hours.

### **Experiment - Controls**

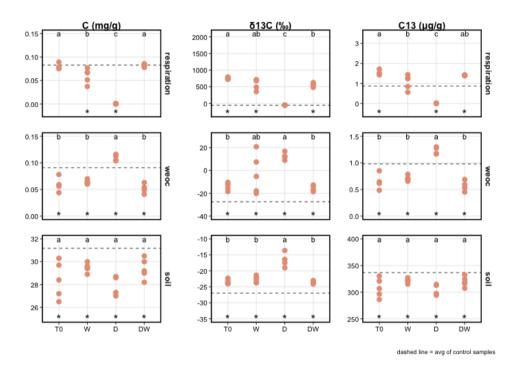


#### **CONTROL FOR PRIMING: solution-C added**

- We had another set of controls to test for priming effects due to the added substrate. labelled substrate was added as a solution
  - 5 g goethite was mixed with the soil,
  - and then 16 mL of the 19.08 % enriched OA solution was added to the soil
- these soils were subjected to only the first two treatments
  - time-zero: jars sealed as soon as goethite was added
  - +C: 16 mL labelled OA solution added
- The jars were sealed for 48 hours.

### Analyses

- 1. **respiration**: headspace samples were collected after the 48-hr incubation. analyzed for CO2 and 13C-CO2
  - LI-7810 (LI-COR), LGR
- 2. **WEOC** (water extractable organic C): organic carbon extracted and analyzed for C content and 13C
  - extracts were dried to powder and analyzed as solid samples
  - VarioIsotope Cube/Isoprime precisION IRMS (Elementar)
- 3. **total soil C**: soil was dried and analyzed for C and 13C content
  - VarioIsotope Cube/Isoprime precisION IRMS (Elementar)

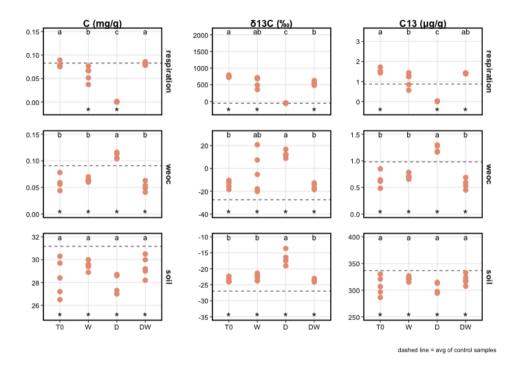


#### **Figure Caption**

total C concentrations, δ13C, and 13C concentrations for T0 (time-zero), W (wetting), D (drying), and DW (drying-rewetting) treatments.

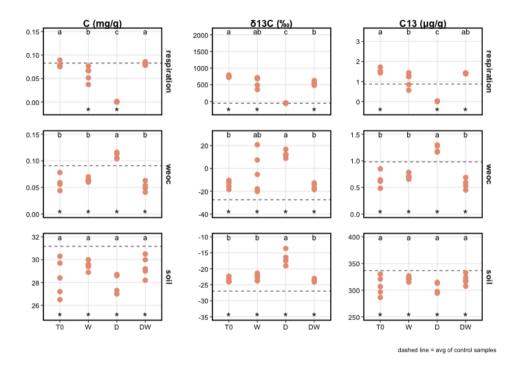
Different letters denote significant differences across treatments.

Asterisks denote significant differences from the unlabelled control samples (dashed line).



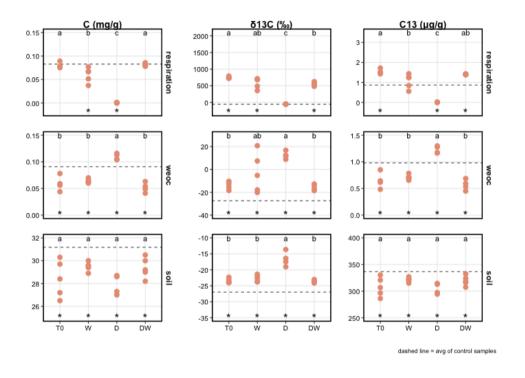
#### respiration

- C amendment did not increase respiration
- in fact, wetting decreased the evolved CO2 by 0.05 mg C/g compared to time-zero
  - negative priming as the oxalic acid was released?
  - high moisture suppressed respiration?
  - lag effect?
- air-dry (D) soils had minimal respiration (as expected)



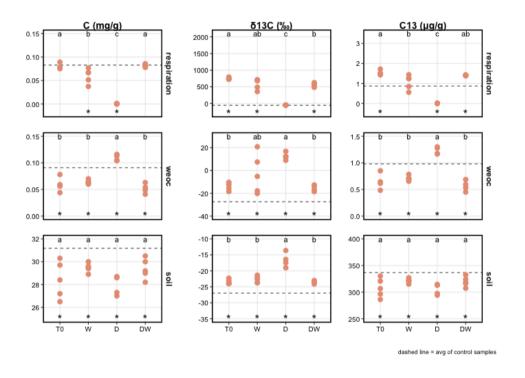
#### respiration

- T-zero, W, and DW soils showed increased 13C enrichment ( $\delta$ 13C) compared to the control soils
  - suggesting that some of the labelled sorbed
     C was desorbed and mineralized
- however, when comparing µg/g of 13C-CO2, only TO and DW were significantly greater than the controls
  - because total CO2-C decreased in W
- thus, the added OA did contribute to the CO2 evolved, but did not increase overall CO2 evolved
  - respiration in these soils is not C limited?
  - no priming effect?
  - perhaps maximum respiration potential was achieved?



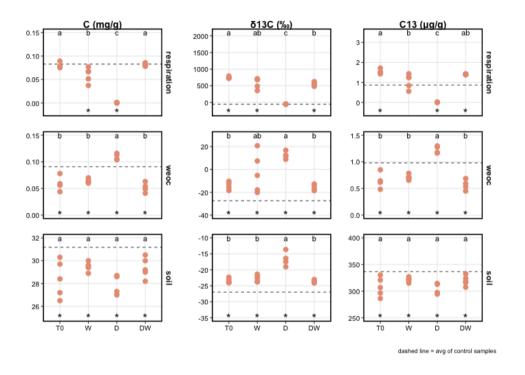
#### respiration

- the enrichment/amount of 13C-CO2 released from the goethite did not differ between TO/W and DW samples.
  - ∘ we expected DW > W
  - W and DW mobilized C equally?
- additionally, W samples showed greater variability than DW samples [STATS NEEDED]
  - preferential wetting patterns?
  - "weaker/non-uniform destabilization" than drying?



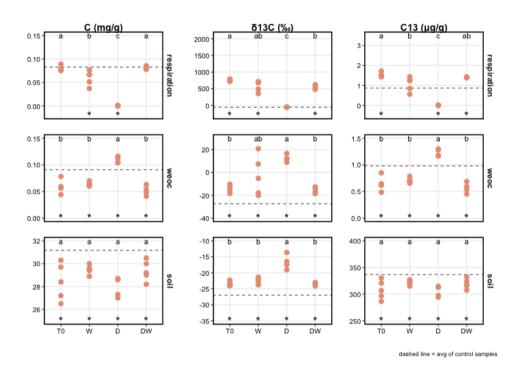
#### **WEOC**

- WEOC concentrations were lower than control for all except D
  - because the C was consumed for respiration?
     (inconsistent with resp data)
- D had the greatest WEOC concentrations
  - (a) desorption of C, (b) microbial necromass/osmolytes, and/or (c) C was not respired and therefore accumulated



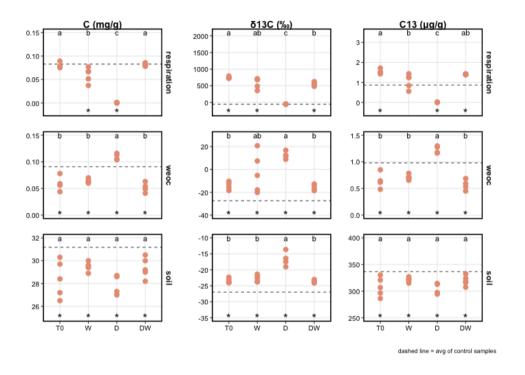
#### **WEOC**

- WEOC was enriched throughout (δ13C)
- W and D were the most enriched (δ13C)
  - suggesting that both wetting and drying caused destabilization of the adsorbed 13C
- however, µg/g 13C was lower than the controls (because total C was also lower than controls)
- air-dry (D) soils had the greatest WEOC-13C
  - because the adsorbed C was destabilized, but not mineralized.
  - When these dry soils were rewet (DW), this available C was mineralized.



#### **WEOC**

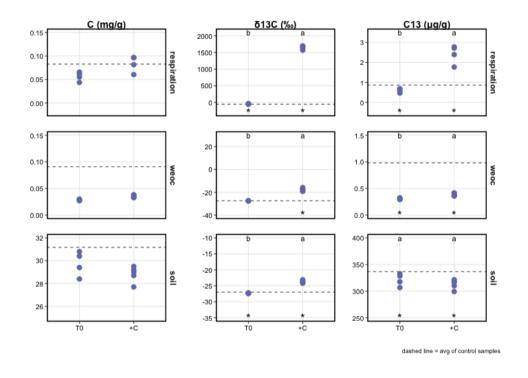
- W  $\delta$ 13C also had greater variability than D/DW (STATS NEEDED)
  - preferential wetting patterns?
  - "weaker/non-uniform destabilization" than drying?



#### soil C

- soil C concentrations were lower than control for all
  - because the C was consumed for respiration?
     (inconsistent with resp data)
- soil C was enriched for all,
- with greatest enrichment in D
  - o ???
  - accumulation of unrespired C?
- however,  $\delta$ 13C did not differ among treatments, and was significantly lower than in control soils
  - o ???

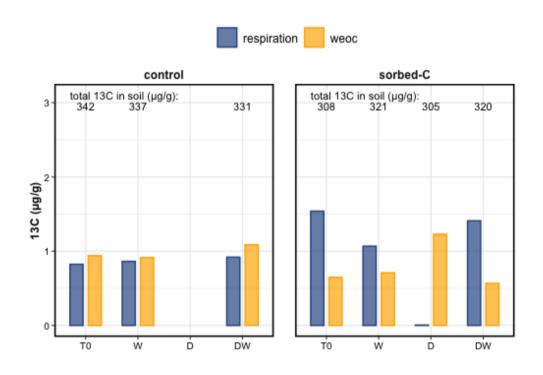
# solution-C (control for priming)



- T0 = goethite mixed with soil
- +C = 13C-OA added in solution form

- respiration, WEOC, and soil were all enriched after addition of soluble 13C-oxalic acid
- respiration: more enriched from solution-C (1648 %) compared to sorbed-C (586 %)
  - OA was more available in solution form than in adsorbed form
- but despite increased OA contribution, overall CO2-C evolved (mg/g) did not increase.

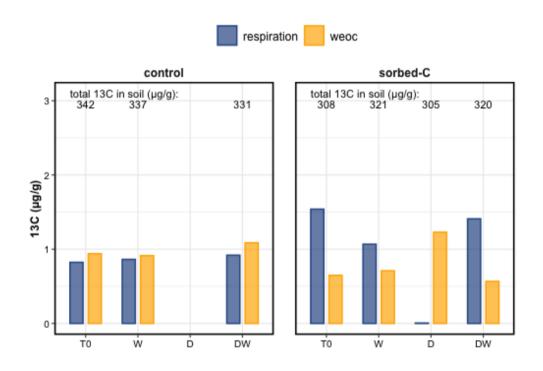
### Mass Balance



#### **SORBED-C**

- these graphs show C13 ( $\mu g/g$ ) in respiration and WEOC fractions for control and sorbed-C samples.
- total C13 in soils was 2 orders of magnitude greater and is not plotted here, but included as text annotation for the respective treatments

### Mass Balance



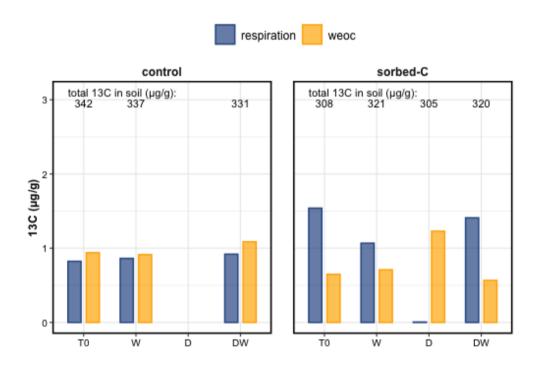
#### **SORBED-C**

- 493 μg (7.72 μg/g) 13C was added in sorbed form.
   can we account for all the 13C added?
- compared to the control soils, the treated soils showed increased C13 (µg/g) in respiration, but decreased C13 in WEOC and total soil:

	resp	soil	weoc
T0	+0.716 *	-34.1 *	-0.291 *
W	+0.205	-15.6 *	-0.204 *
D	NA †	NA †	NA †
DW	+0.490 *	-11.3 *	-0.520 *

t no control samples for comparison asterisk = significant difference from control

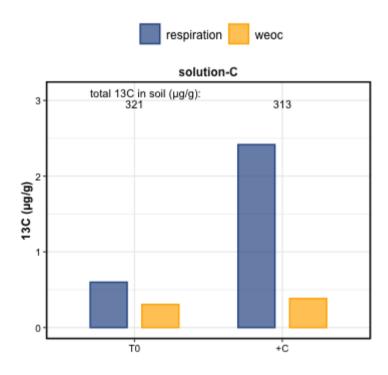
### Mass Balance



#### **SORBED-C:** isotope retention

- 493 μg (7.72 μg/g) 13C was added in sorbed form.
- but there was a net decrease of 13C in the treated soils, comparing 13C added with (resp + soil)
- is there a missing piece?
  - methane? no, because CH4 production was very, very low, and did not change with treatment/OA addition.
  - spatial variability? power analysis indicated that minimum detectable change in soil was 12.00 μg/g.

### Mass Balance: solution-C



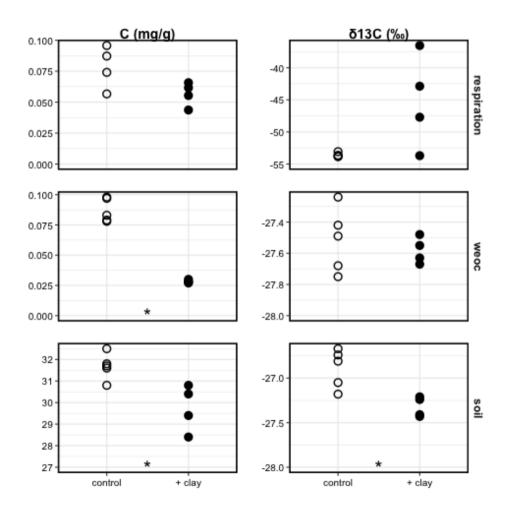
384  $\mu$ g (6.02  $\mu$ g/g) 13C was added as solution

- compared to T0, the +C soils showed:
  - small but significant increase in WEOC-13C
  - small but significant increase in resp-13C
  - no significant change in soil-13C
     SO WHERE DID THE 13C GO???

	resp	soil	weoc
+C	+1.82 *	-8	+0.08 *

- spatial variability of soil?
  - power analysis of T0 vs. +C soil data indicated a minimum detectable change of 22.89 μg/g C13

### extra: How did clay addition influence C?



We compare time-zero of control (soil only) with time-zero of solution-C treatment (soil + goethite)

- respiration did not change significantly, but enrichment was more variable
  - o ???
- weoc decreased, but enrichment did not change
  - previously available SOM was now sorbed onto goethite
- soil C decreased, as did enrichment
  - o ???

# Conclusions

coming soon

### **Session Info**

Date run: 2021-02-03

#### Slides prepared in RMarkdown using xaringan

```
## R version 4.0.2 (2020-06-22)
## Platform: x86_64-apple-darwin17.0 (64-bit)
## Running under: macOS Catalina 10.15.7
## Matrix products: default
## BLAS: /System/Library/Frameworks/Accelerate.framework/Versions/A/Frameworks/vecLib.framework/Versions/A/libBLAS.dylib
## LAPACK: /Library/Frameworks/R.framework/Versions/4.0/Resources/lib/libRlapack.dylib
## locale:
## [1] en_US.UTF-8/en_US.UTF-8/en_US.UTF-8/C/en_US.UTF-8/en_US.UTF-8
## attached base packages:
                graphics grDevices utils
## [1] stats
                                             datasets methods base
## other attached packages:
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                                                      PNWColors_0.1.0 drake_7.13.0
                                                                                     forcats_0.5.1
## [7] stringr_1.4.0 dplyr_1.0.3
                                      purrr_0.3.4
                                                      readr 1.4.0
                                                                     tidvr 1.1.2
                                                                                     tibble 3.0.6
## [13] ggplot2 3.3.3 tidyverse 1.3.0
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