Permanganate oxidizable carbon for soil health: Does drying temperature matter?

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Abstract

Soil health assessments evaluate and monitor the effects of conservation management on soil properties. A popular measurement, permanganate oxidizable carbon (POXC), is routinely included in these assessments. The standard POXC protocol calls for air-dried soil samples, but commercial laboratories typically dry samples in a forced-air oven before routine analysis. In order to evaluate if heat would change POXC measurements, we treated soil (52 silty clay loam and 51 sandy loam samples) with oven drying at 45 and 65 °C and compared their POXC values with those from air-dried samples. We also examined relationships between POXC values and soil organic matter, pH, and electrical conductivity across drying treatments. Drying soil did not substantially change POXC values for either soil texture and did not change the relationships between POXC and other measured soil properties. This work suggests that commercial laboratories could perform POXC analysis on soil samples dried using heat.

1 INTRODUCTION

Labile fractions of soil organic matter are popular soil health indicators (Nunes et al., 2020), favored for their responsiveness to soil management and their ties to soil microbial activity (Culman et al., 2012; Hurisso et al., 2016; Morrow, Huggins, Carpenter-Boggs, & Reganold, 2016). Permanganate oxidizable carbon (POXC; Weil, Islam, Stine, Gruver, & Samson-Liebig, 2003) measurements represent active carbon in suites of soil health tests (Andrews, Karlen, & Cambardella, 2004; Moebius-Clune et al., 2016). This method is currently being used in national soil health assessments (e.g., Norris et al., 2020) and in research studies that address soil health response to conservation management practices.

The procedure developed for measuring POXC in soil (Weil et al., 2003) specifies the use of air-dried soil. This recommendation is based on a comparison of POXC values in sandy loam and clay loam soils subjected to different passive drying treatments. For samples assessed in the Weil et al. (2003) study, oxidized carbon increased, and measurement variability decreased, with increasing soil dryness, which was attributed to higher redox potential of soils that had been dried for a longer period of time (24 h). The authors acknowledged that drying conditions (humidity and temperature) may influence carbon oxidation and that standardized soil drying conditions should be considered when making POXC comparisons.
If different soil drying conditions can introduce variability in POXC measurements, we might expect the relationships between POXC and other soil properties to shift as well. Soil texture, organic matter content, pH, and electrical conductivity (EC) are routinely measured on soil samples that are submitted to laboratories for soil testing services. Many studies have reported a strong positive relationship between POXC and soil organic carbon (Calderón et al., 2017; Culman et al., 2012; Hurisso et al., 2016; Lucas & Weil, 2012; Morrow et al., 2016; Nunes et al., 2020; Veum, Goyne, Kremer, Miles, & Sudduth, 2014), and this relationship holds across a variety of soil textures, chemistries, and management systems. Furthermore, Culman et al. (2012) and Hurisso et al. (2016) demonstrated that inclusion of soil particle size into predictive models does little to increase the amount of variability explained between POXC and soil organic carbon. The relationships between POXC and inorganic soil chemical properties (including pH and EC) have not been widely explored; however, Hurisso, Culman, and Zhao (2018) reported a positive correlation between POXC and pH in soils from three Ohio fields.

The standard procedure of air drying prior to POXC analysis is cumbersome for commercial laboratories due to space and time requirements. Almost all analyses performed in soil testing laboratories use soil dried by hot forced air (35–65 °C; see North Central Region, 2011; Texas A&M Extension, 2012; Zhang & Henderson, 2018). If active carbon is to be provided as a routine commercial soil test, it is worthwhile to understand if POXC measurements are affected by heat and drying in a forced air oven. The objective of this study was to assess if drying in an oven influences POXC measurements of soils across two textural classes (silty clay loam and sandy loam) and if the drying treatment altered the relationship between POXC and other commonly measured soil properties. To meet this objective, we examined POXC measurements on soils that had been air dried, dried in an oven at 45 °C, or dried in an oven at 65 °C.

2 METHODS

Soil samples used for this study were collected from annual cropping fields in southeastern North Dakota, USA, in 2014 (Butcher, Wick, DeSutter, Harmon, & Chatterjee, 2018). A total of six fields were sampled, each hosting five transects that paralleled the crop rows. Four soil samples were collected (0–15 cm depth) from each transect at 25-m spacing. Soil samples were measured for soil particle size distribution by the hydrometer method (Gee & Bauder, 1986) and soil pH and EC of 1:1 soil/water (Rhoades, 1996; Thomas, 1996). Soil from three fields was classified as silty clay loam texture and had Zea mays L. crops, and soil from three fields was classified as sandy loam texture and had Glycine max (L.) Merr. crops. Soil samples were air-dried, sieved to 2 mm, and stored at room temperature until 2018. Gravimetric water content was measured on a subsample of the air-dried soils (Gardner, 1986) and ranged from 0.01 to 0.04 g water g⁻¹ soil. A second subsample was analyzed for soil organic matter percentage based on loss-on-ignition (Nelson & Sommers, 1996).

For the experiment, soil samples were split into three drying treatments prior to POXC analysis: air-dried (processed directly from archive), dried at 45 °C in a forced-air oven, or dried at 65 °C in a forced-air oven. After treatment, soils were analyzed for POXC according to Weil et al. (2003) on triplicate 2.5-g samples. Analytical triplicates were averaged to obtain a single POXC value for each sample by drying treatment. In some cases, there was not sufficient soil to complete all analyses, so after removing samples with incomplete data, we obtained a total of 103 samples, with 52 silty clay loam samples and 51 sandy loam samples.

To assess if drying treatment influenced the POXC measurements, and if it was texture-dependent, we compared mean POXC within soil textural classes across drying treatments using a Kruskal–Wallis test with Dunn’s multiple comparison post-hoc tests. These are nonparametric means comparison tests that allow for sample dependency across treatments. To assess if drying treatments influenced the relationship between POXC and soil organic matter, we fit simple linear regression models to all observations as well as separated by textural class. We also assessed relationships between POXC and pH (5.95–8.30 range) and EC (0.11–3.89 dS m⁻¹ range). All data analysis and visualization were conducted in R (R Core Team, 2020) with assistance from ‘dplyr’ (Wickham, Francois, Henry, Muller, & Studio, 2020), ‘FSA’ (Ogle, Wheeler, & Dinno, 2020), ‘pastecs’ (Grosjean, Ibanez, & Etienne, 2018), and ‘scales’ (Wickham, 2019) packages.
3 | RESULTS AND DISCUSSION

Our goal was to assess if drying treatments produced different POXC measurements for two soil textures. According to summary statistics of the measurements, we did not observe strong effects of temperature on POXC measurements. The POXC values in each soil texture were normally distributed, as indicated by similar means and medians that fell in the middle of the range in data values for each drying treatment (Figure 1). These distributions were stable across drying treatments. The mean POXC values in the silty clay loam soil samples did not differ (air-dried = 671, 45 °C = 677, and 65 °C = 668 mg C kg⁻¹ soil), while the mean POXC values in the sandy loam soils decreased from 601 mg C kg⁻¹ soil when air-dried to 577 mg C kg⁻¹ soil at 45 °C to 557 mg C kg⁻¹ soil at 65 °C (Figure 1).

The range of POXC values for these soil samples was intermediate (about 400–900 mg C kg⁻¹ soil) compared with ranges reported in surveys of many soil types (0–1,500 mg C kg⁻¹ soil, Calderón et al., 2017; Culman et al., 2012; Hurisso et al., 2016). In studies that identified POXC as a reliable indicator for detecting differences in soil health across management systems, a significant treatment difference coincided with a change in POXC of about 100 mg C kg⁻¹ soil or greater (Culman, Snapp, Green, & Gentry, 2013; Diederich, Ruark, Krishnan, Arriaga, & Silva, 2019; Ghimire, Ghimire, Mesbah, Sainju, & Idowu, 2019; Lucas & Weil, 2012; Morrow et al., 2016; Singh et al., 2020).

Because the oven drying did not cause this large of a shift in mean POXC, we do not believe that oven-drying these soil samples would change the interpretation of the analysis or the utility of POXC as a soil health indicator.

We were also interested in evaluating if drying treatments altered the relationship between POXC and other soil properties, including soil organic matter content (as measured by loss-on-ignition), which is more common than soil organic carbon as a routine commercial soil test. Relationships between POXC and pH and POXC and EC were weak (R² values ranged from .01 to .06 for pH and <.01 to .13 for electrical conductivity) and did not change substantially with soil drying. The relationships between POXC and pH were not significant for either soil texture in any drying treatment, but the combined model was significant for soils dried at 65 °C (POXC = 63 × pH + 113, R² = .04, p = .03). Combined models for POXC and EC were not significant. For the silty clay loam soil, relationships for air-dried soil (POXC = −40 × EC + 712, R² = .13, p = .01) and soil dried at 65 °C (POXC = −31 × EC + 700, R² = .08, p = .05) were significant. For the sandy loam soil, only the soil dried at 65 °C had a significant relationship between POXC and EC (POXC = 53 × EC + 522, R² = .08, p = .04).

In general, the relationships between POXC and soil organic matter content were linear and stable across drying treatment and across soil textures (Figure 2). Simple linear regression model parameters were similar (as indicated by overlap of parameter 95% confidence intervals) between models that included both soil textures, models for each soil texture, and models across drying treatments (Figure 2). Arguably, increased drying temperature slightly decreased residual variation (indicated by increases in the coefficient of determination, or R²) between soil organic matter content and POXC.

On the basis of these observations, we conclude that drying soil in a forced-air oven at either 45 or 65 °C did not alter POXC measurements on two soil textures, nor did it alter the relationship between POXC and soil organic matter measurements. We did not observe strong relationships between POXC and pH or EC at any drying temperature. It is important to note that all samples used in this analysis had previously been air dried, and the effect of drying with heat should also be evaluated on field-moist soils. However, these results indicate that POXC analysis may be performed on soil samples that have been oven dried without strong effects on POXC measurements and data interpretation.

Because soil health assessment appears to be on the rise, it is important that laboratories use standardized protocols to allow valid comparisons across projects and data sets. The POXC method is subject to sources of variability that relate to soil handling (Wade et al., 2020), but overall, the method appears to be fairly robust across different soil types and slight variations in sample handling. Certainly, these details are important when considering the robustness of soil health indicators, including POXC.
Fitted linear regressions between soil organic matter (SOM %, based on loss-on-ignition) and permanganate oxidizable carbon (POXC, mg C kg$^{-1}$ soil) after three soil drying treatments: air-dried, drying in an oven at 45 °C, or drying in an oven at 65 °C. Plotted regression lines (with 95% confidence intervals) represent the fitted model for all soil samples ($n = 103$), which includes silty clay loam soils ($n = 52$, blue symbols) and sandy loam soil ($n = 51$, red symbols). Fitted regression parameters for the overall model, as well as models for each textural class are provided, with 95% confidence intervals (95% CI) on each model parameter, for each drying treatment.

| Textural Class     | Model Parameters          | Slope 95% CI  | Intercept 95% CI | $R^2$ | p-value |
|--------------------|---------------------------|---------------|------------------|-------|---------|
| Overall            | POXC = 96 * SOM + 274     | (80, 113)     | (211, 338)       | 0.57  | <0.001  |
| Air-dried          | POXC = 112 * SOM + 204    | (98, 126)     | (150, 258)       | 0.71  | <0.001  |
| 45 °C              | POXC = 117 * SOM + 171    | (104, 131)    | (119, 223)       | 0.75  | <0.001  |
| 65 °C              | POXC = 138 * SOM + 78     | (99, 177)     | (-90, 247)       | 0.50  | <0.001  |
| Silty clay loam    | POXC = 149 * SOM + 33     | (115, 184)    | (-117, 184)      | 0.60  | <0.001  |
| Air-dried          | POXC = 145 * SOM + 42     | (108, 183)    | (-120, 205)      | 0.55  | <0.001  |
| 45 °C              | POXC = 128 * SOM + 189    | (100, 156)    | (97, 281)        | 0.63  | <0.001  |
| 65 °C              | POXC = 128 * SOM + 166    | (103, 152)    | (86, 245)        | 0.69  | <0.001  |
| Sandy loam         | POXC = 129 * SOM + 143    | (107, 150)    | (73, 212)        | 0.75  | <0.001  |

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AUTHOR CONTRIBUTIONS
Conceptualization: SM, TD; Formal analysis: CG; Investigation: AD, KB; Resources: CG; Supervision: CG, SM, TD; Visualization: CG; Writing–original draft: CG; Writing–review and editing: CG, AD, SM, KB, TD.

CONFLICT OF INTEREST
The authors declare no conflict of interest.

DATA AVAILABILITY
These data are available from the authors upon request.

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