Research Square

Preprints are preliminary reports that have not undergone peer review.
They should not be considered conclusive, used to inform clinical practice,
or referenced by the media as validated information.

Accuracy, precision, and repeatability of loss-on-ignition protocols for estimating soil organic carbon relative to dry combustion

Luis Bentancor

lbent@fagro.edu.uy

University of the Republic

Lee Burras

Iowa State University

Michael Thompson

Iowa State University

Peter O'Brien

National Laboratory for Agriculture and the Environment

Bradley Miller

Iowa State University

Research Article

Keywords: soil organic matter, soil organic carbon, conversion equations, loss on ignition, dry combustion

Posted Date: March 2nd, 2026

DOI: https://doi.org/10.21203/rs.3.rs-8752000/v1

License: This work is licensed under a Creative Commons Attribution 4.0 International License.
Read Full License

Additional Declarations: No competing interests reported.

Abstract

Soil organic matter (SOM), including its primary constituent organic carbon, is fundamental to soil health and a major component of the global carbon cycle. Reliable assessment of SOM stocks requires precise, repeatable measurements and sufficiently large sample sizes to capture spatial heterogeneity and detect slow temporal change. Loss-on-ignition (LOI) provides a low-cost approach for increasing sample throughput, yet the use of multiple LOI protocols has limited comparability across studies and obscured understanding of associated measurement error. This study evaluated three LOI methods for estimating SOM and predicting soil organic carbon (SOC) measured by dry combustion (DC): ignition at 360°C for 2 h (LOI360), 400°C for 16 h (LOI400), and 550°C for 3 h (LOI550). Linear conversion equations were developed to relate LOI-derived SOM to DC-SOC, and model performance was assessed using R^2, RMSE, MAE, MedAE, and Lin's concordance correlation coefficient. LOI360 and LOI400 produced comparable accuracy and precision in predicting SOC, but LOI400 exhibited the strongest repeatability. LOI400 also provided an effective balance between incomplete SOM combustion at lower temperatures and mineral mass loss at higher temperatures, whereas LOI550 performed poorly due to variable mineral decomposition. Localized conversion equations grouped by landform region did not substantially improve SOC predictions relative to a single generalized equation. Although LOI400 showed greater measurement variability than DC, its low cost enables larger sample sizes, thereby improving characterization of spatial SOC variability. Using the generalized equation SOC = 0.365 * LOI400 − 0.039, LOI400 represents a reliable and economical alternative to DC for SOC estimation in resource-limited settings.

1. Introduction

Beyond the environmental and agronomic benefits of soil organic matter (e.g., water retention and nutrient storage), the carbon component of soil organic matter is a large reservoir in the global carbon cycle [1]. With high interest in monitoring soil carbon stocks and potentially paying landowners or land managers for carbon sequestered in their soil, a fast, cost-effective, and precise method for measuring soil organic carbon is needed. Using soil organic matter (SOM) measurements to estimate soil organic carbon (SOC) content is a cost-effective and straightforward approach. However, a conversion factor is required [2] .

For 135 years, the "van Bemmelen factor" has served as the standard for converting SOM measurements to SOC, with the assumption that SOM contains a mean of 58% SOC [3]. Based on an exhaustive review of literature from the 1820s to the 2000s, [2] concluded that the "van Bemmelen factor" is overly high. This study found a median value of 53% for empirical observations and 50% based on theoretical estimations. Consequently, instead of employing the [3] conversion factor of 0.58 kg SOC / kg SOM to produce an estimate of soil organic carbon from loss-on-ignition (LOI-SOC), [2] recommended using 0.53 or 0.50 kg SOC / kg SOM, considering the empirical or theoretical estimations, respectively.

High-temperature dry combustion (DC) has become the standard method for direct SOC (DC-SOC) determination due to its high precision [4]. Moreover, depending on the instrument used, nitrogen (N) and sulfur (S) can be measured simultaneously, and DC may be integrated with a mass spectrometer for stable isotope analysis [5]. However, DC has some disadvantages. For accurate C and N determination, the sample must be finely ground and well homogenized to ensure complete combustion and representative results. While grinding to ≤ 2 mm is standard for most soil analyses, finer grinding may be required for DC to minimize variability caused by localized organic matter concentrations or incomplete oxidation [6]. A more significant issue, for both cost and accuracy of the analysis, is that pretreatment is usually required for samples containing inorganic carbon (IC). Other drawbacks of this method include equipment costs, which range from $40,000 to $50,000, and operational costs, which encompass high electricity consumption and the use of high-purity gases [5, 7].

Loss on ignition (LOI) is a commonly employed technique for determining SOM concentrations (LOI-SOM; [8]). For example, LOI-SOM is already included in the standard soil fertility analysis of many commercial labs. This approach has been reported to be a reliable and inexpensive method for predicting SOC using a standard conversion such as the van Bemmelen factor [9]. [10] compared LOI using 550°C for 3 hours to DC, concluding that DC operating costs were 12 times more expensive and the process took 2.3 times longer to complete. Like DC, LOI has the advantage of not generating hazardous waste, which is a disadvantage for older methods, such as the Walkley-Black wet-combustion method, which produces chromium (Cr) as a byproduct [11]. Nonetheless, a universally standardized protocol for LOI has been elusive due to many factors that may affect the measurement, including soil mineralogy, calcium carbonate content, heating temperature, and time exposed to that heating temperature.

Although LOI methods have been published in laboratory methods standards manuals (e.g., [12]), the literature lacks consensus regarding the optimal temperature and ignition duration for the LOI method in measuring SOM. Temperatures ranging from 300 to 850°C and durations from 2 to 28 hours have been suggested. Low temperatures of ignition over long durations (e.g., 375°C for 16 hours) have been reported to be more accurate for measuring SOM than high temperatures over short periods (e.g., 850°C for 0.5 hours) due to incomplete oxidation of C for short time periods or due to mass loss from minerals at high temperatures [13] .

Many variations of LOI methods have been developed and reported. For example, using 430°C for 24 hours, [14] observed no overestimation of organic matter concentration due to the presence of calcium carbonate. He recommended temperatures between 375 and 450°C for both calcareous and non-calcareous soils, but not for soils with gibbsite, where the temperature should be reduced to 300°C. On the other hand, [8] concluded that a temperature of at least 550°C for 3 hours should be used to completely oxidize the organic material. However, they also recommended using a clay correction factor to offset overestimation caused by mineral mass loss.

[11] also reported that the optimal heating temperature and time depend on the mineralogy of the sediment source. For example, CO_2 can be volatilized from siderite, dolomite, and rhodochrosite at 425°C [15, 16]. Goethite (FeOOH) can be dehydroxylated between 280 and 400°C [17], while gibbsite ($Al(OH)_3$) loses an important quantity of mass around 300°C [14]. Similarly, [18] reported that the water loss from Fe or Mn oxyhydroxides significantly affected LOI results.

Among the layer silicate clay minerals, kaolinite loses about 14% of its mass by dehydroxylation at temperatures between 400 and 600°C, with the greatest loss around 500°C [19, 20]. Dehydration by evaporation of interlayer water in vermiculite and smectite occurs at temperatures greater than 250°C. The 2:1 layer silicate minerals (e.g., illite, chlorite, vermiculite, and smectite) lose about 5% of their mass by dehydroxylation between 500 and 900°C [17, 19–21]. For non-layer silicate minerals, mass losses of 15.84%, 2.45%, and 2.02% have been reported when using LOI at 550°C for gypsum, goethite, and dolomite, respectively [22]. These authors suggested a correction for mass loss due to dehydration and dehydroxylation when working with subsurface samples - where organic C concentration is likely to be low - or soils containing appreciable quantities of goethite, kaolinite, or vermiculite. However, accurate correction requires mineral abundance data, which are typically more expensive to obtain on a per-sample basis than conducting DC analyses. Table 1 summarizes the known temperature thresholds for mass loss from soil components other than organic matter.

Table 1
Summary of minerals observed to lose mass in the range of temperatures used in LOI methods [17, 19–21, 23].

Mineral Type	Name	Key Temperature Threshold
1:1 layer silicate	Kaolinite	Dehydroxylation 400–600°C (~ 14%)
2:1 layer silicate	Chlorite	Dehydroxylation 500–900°C (~ 5%)
2:1 layer silicate	Illite	Dehydroxylation 500–900°C (~ 5%)
2:1 layer silicate	Smectite	Dehydration at 250°C; Dehydroxylation 500–900°C (~ 5%)
2:1 layer silicate	Vermiculite	Dehydration at 250°C; Dehydroxylation 500–900°C (~ 5%)
Carbonate	Calcite	CO_2 volatilized at 675°C
Carbonate	Dolomite	CO_2 volatilized at 425°C; Mass loss at 550°C (~ 2%)
Carbonate	Rhodochrosite	CO_2 volatilized at 425°C
Carbonate	Siderite	CO_2 volatilized at 425°C
Sulfate	Gypsum	Mass loss at 550°C (~ 16%)
Aluminum hydroxide	Gibbsite	Dehydroxylation at 250–300°C (31%)
Iron Oxyhydroxide	Goethite	Dehydroxylation 280–400°C (10–11%)
Tectosilicate	Feldspar	None
Tectosilicate	Quartz	None
Phyllosilicates	Mica	None

The approach for converting SOM to SOC depends on the goal. The conversion factors of [3] and [2] assume that the conversion should be based on the proportion of organic carbon contained in organic matter. However, other factors – such as the mass loss from clay minerals – introduce additional variables to the relationship between laboratory methods. This additional complication suggests that a conversion equation, rather than the proportional composition of C in SOM, should be applied to more accurately relate the results of one method to another. [24] recommended using linear equations to convert LOI-SOM to DC-SOC. The high coefficient of determination (R^2) results for their linear equations supported the inclusion of an intercept to improve conversion precision. In addition, a Tukey's test for significant differences between the slopes and intercepts of equations for contrasting Major Land Resource Areas (MLRA) suggested the predictive equations should be locally customized. However, a

generalized conversion equation encompassing the pooled data was not tested against the localized conversion equations. Considering that the resources to measure both LOI-SOM and DC-SOC to create customized equations for every locality might often be impractical, determining the precision of a generalized equation would help assess the suitability of LOI for SOC change detection.

Further complicating the use and comparison of LOI measurements is the diversity of method parameters employed, which has contributed to LOI being perceived as a method with low precision for predicting SOC. Various factors may influence LOI-SOM results under different methods; therefore, measurements of SOM with varying temperatures and durations are unlikely to be directly comparable. This study seeks to disambiguate the performance of LOI by testing three methods: heating for two hours at 360°C [9], heating for sixteen hours at 400°C [12], and heating for three hours at 550°C [8]. Comparison of these methods among a range of soil samples enabled: 1) assessment of the relationships between DC and the different LOI methods, 2) examination of the variability and sources of error in converting SOM to SOC by LOI treatment, and 3) evaluation of the repeatability (i.e., measurement error) of the different LOI methods compared to DC.

2. Materials and Methods

2.1. Study area and soil samples

A collection of 424 samples was assembled to represent a diversity of soil conditions from four distinct landform regions across Iowa, USA (Fig. 1). These regions encompass a diverse range of glacial landscapes, characterized by contrasts in parent material, mineralogy, and topography (Table 2). Land use in these areas is predominantly corn and soybean production. One extreme outlier with consistently high values across all LOI treatments was excluded to avoid biasing model evaluation, resulting in a final dataset of 423 samples.

Table 2

Descriptions of soil landscapes where soil samples were collected and analyzed for this study. Note that all minerals exhibit spatial variability across landscapes and with depth. The presence of calcite in this study was identified in individual samples by testing soil pH.

Landform Region[a]	Dominant Parent Material	Clay mineralogy [26, 27]	Landscape	Example Soil Series & Taxonomic Subgroup
DML	Clay loam, calcareous till	Calcite, goethite, smectite, and mica; Smaller amounts of kaolinite and hydroxyl-interlayered minerals	Closed basins with low relief	Clarion and Canisteo (Udolls – Aquolls)
IES	Silty clay loam, Peoria loess (variable thickness or absent) mixed with or over Pre-Illinoian till	Calcite and feldspar; some mica; variable illite and kaolinite	Broad dissected plains	Kenyon and Clyde (Udolls – Aquolls)
SIDP	Silty clay loam, Peoria loess (~ 3 m thick) over Pre-Illinoian till	Calcite and smectite; variable illite and kaolinite	Dissected hills and flats	Marshall and Tama (Udolls)
NWP	Silty clay loam, Peoria loess (~ 5 m thick) over Pre-Illinoian till	Calcite, feldspar, smectite, variable illite, and kaolinite	Gently rolling hills	Galva and Sac (Udolls)

[a] DML = Des Moines Lobe; SIDP = Southern Iowa Drift Palin; IES = Iowa Erosion Surface; NWP = Northwest Plain.

All samples were analyzed for soil texture class and pH. All samples with a pH greater than seven were tested for the presence of inorganic carbon. All locations included surface horizon samples to a depth of 20 cm. Of those, 79% were collected using a truck-mounted hydraulic soil sampler (Giddings Machine Company, Windsor, CO) to a depth of 200 cm. Of the total samples, 50% corresponded to A, 35% to B, and 15% to C horizons. This distribution of horizons included a range of SOM concentrations and textural classes (Fig. 2).

Particle size analysis (PSA) was conducted using laser diffraction with a Malvern Mastersizer 3000 and Hydro EV attachment. Samples were oven-dried at 65°C for 24–48 hours, depending on initial moisture content and sample texture. After drying, the samples were ground and sieved to a 2 mm mesh size and homogenized by passing the sample through a Haver RT 12.5 mm splitter three times. The Malvern Mastersizer 3000 passes laser pulses through a water suspension of soil particles. The laser's interaction with the particles alters the angle of the light, which is measured by the instrument. Mie diffraction theory determines the equivalent spherical diameter of the soil particles (Malvern Instruments, 2017). Pre-treatment of samples for PSA and quality control protocols were conducted per [28].

2.2. Lab analysis

2.2.1. Dry combustion

DC-SOC was treated as the standard reference method for accurately measuring SOC, which was calculated as the difference between total carbon and IC. Solid samples ground to < 0.25 mm were dried at 105°C before being weighed for analysis. Then, 30 mg of these samples were flash combusted in an oxidative environment to determine the total carbon, producing carbon dioxide from the carbon compounds in the samples [29]. Carbon dioxide (CO_2) was quantified using a thermal conductivity detector (FlashSmart Elemental Analyzer, Thermo Fisher Scientific, Waltham, MA, USA). The instrument was calibrated before each analytical sequence using five atropine standards to generate the first-order calibration curve. Calibration was accepted with an $R^2 > 0.999$. Three reference soil standards were analyzed following calibration. To proceed with sample analysis, these samples were required to agree within ± 3 standard deviations of the reference values. For quality control, one duplicate experimental sample and one laboratory control sample were included for every ten experimental samples. Duplicate sample data were acceptable if within ± 10% relative difference (RPD).

Inorganic carbon was analyzed using a pressure calcimeter to measure the pressure increase from CO_2 gas evolution after adding acid to soil samples containing carbonate minerals. Eleven soil standards amended with calcium carbonate, ranging from 0-2.4% IC, were used to generate a daily, linear calibration curve with an $R^2 > 0.995$. Two check samples of reference calcareous soil standards were run with each set of calcareous samples and were required to agree within ± 10% of the known reference value to be acceptable [30].

Quality control for IC determination included one duplicate sample for every ten experimental samples. Three laboratory control samples of reference calcareous soil standards and one laboratory blank were run with every set. Acceptable laboratory control data had a recovery within ± 3 SD of the expected mean IC percentage, and the laboratory blank was accepted if the area counts were less than the area counts of the lowest standard. If the blank and the reference sample analyses fell outside the expected ranges, the entire run was repeated.

2.2.2. Loss on ignition

All LOI-SOM treatments were run using a muffle furnace (Lindberg 51000 Series, model 51442). Sixty-two Fisher brand FB-965-E crucibles were filled with 8 grams of < 2-mm oven-dry soil per run. Before furnace ignition, samples were oven-dried at 105°C overnight, cooled in a desiccator, and weighed to record the mass to 0.01 g. Three treatments were applied to assess the effect of duration and temperature of ignition. For treatments LOI360, LOI400, and LOI550, the temperature of ignition and duration were two hours at 360°C [9], sixteen hours at 400°C [12], and three hours at 550°C [8], respectively. LOI-SOM calculations were performed using Eq. (1), where ODSM and ISM represent the oven-dry soil mass and ignited soil mass, respectively.

$$LOI\ \left(g\,Kg^{-1}\right) = \frac{(ODSM - ISM)}{ODSM}\ \times\ 1000 \qquad (1)$$

2.3. Statistical analysis

2.3.1. Defining precision, accuracy, and repeatability

The terms precision, accuracy, and repeatability, among others, are often conflated or have different meanings in various contexts. This research was a comprehensive exercise in all three aspects because the process included evaluating the conversion equations' ability to represent patterns in the data (goodness-of-fit), how closely those conversion models matched the target variable, and the variability stemming from measurement error. High precision indicates relationships between variables are consistent (low dispersion), while high accuracy indicates that predictions are close to the reference values (unbiased) (Fig. 3). Repeatability in this context is constrained to the variability observed when measuring the same sample multiple times.

2.3.2. Precision

Traditionally, the strength of equations describing observed relationships between SOM and SOC has been assessed with R^2. However, our analysis requires comparison of multiple sample sets with different variances, and R^2 is explicitly based on variance. In the R^2 Eq. (2), the residuals from the mean model are compared to the residuals of the prediction model being tested (3).

$$R^2 = 1 - \frac{SS_{residual}}{SS_{total}} \qquad (2)$$

where SS_{total} is the total sum of squares, or the total variance (3), and $SS_{residual}$ is the residual sum of squares, or the unexplained variance (4).

$$SS_{total} = \sum \left(y_i - \bar{y_i}\right)^2 \qquad (3)$$

$$SS_{residual} = \sum \left(y_i - \hat{y}_i\right)^2 \qquad (4)$$

where y_i are the observed values, $\hat{y}_i$ are the predicted values, and $\bar{y_i}$ is the sample mean.

R^2 values for sample sets with low variance will tend to be low because there is little variance to be explained. Conversely, when there is more variance to explain, R^2 values will tend to be higher. Given the nature of R^2, smaller sample sets would have a higher potential for inflated R^2 values. Therefore, R^2 is used to assess the strength of the relationship between DC-SOC and LOI-SOM, thereby evaluating the reliability of individual conversion equations. However, when determining the optimal method for converting LOI-SOM to a DC-SOC standard, accuracy is the key consideration.

2.3.3. Accuracy

The primary goal of a conversion equation is to yield results that accurately match the values obtained by the reference method. To assess the performance of a conversion equation, predictions (i.e., LOI-SOC) should be compared with observed (i.e., DC-SOC) values and evaluated using metrics of deviation from the target values. Also, given the limitations of metrics with high dependencies on variance, assessing model performance solely through R^2 may be misleading. Moreover, a single metric reflects only one dimension of a model's errors, highlighting just a specific facet of the interaction between the model and data [31]. As measures of accuracy, the two most widely used error metrics were adopted: root mean square error (RMSE) (5) and mean absolute error (MAE) (6) [32].

$$RMSE = \sqrt{\frac{SS_{residual}}{n}} \qquad (5)$$

$$MAE = \frac{1}{n}\sum_{i=1}^{n} |SS_{residual}| \qquad (6)$$

According to [31], RMSE is particularly appropriate when residuals are expected to be unbiased and normally distributed, as it disproportionately penalizes large errors and is sensitive to variance. This sensitivity makes RMSE effective for identifying models that produce occasional large deviations, especially when such deviations are relevant to the model's use case. In contrast, MAE treats all errors equally, providing a straightforward and robust measure to summarize error magnitude.[33] advocate for MAE when the goal is to assess general predictive accuracy without inflating the influence of outliers or assuming normality. MAE is more interpretable in contexts where the frequency rather than the magnitude of large deviations is more relevant to model performance.

Because these accuracy metrics are influenced by the shape of the residual distribution, the skewness and kurtosis of the errors were also calculated in R using the moments package [34]. Skewness evaluates the symmetry of the residuals, with values near zero indicating a balanced distribution. A positive skew suggests underestimation (long right tail), while a negative skew indicates overestimation (long left tail). On the other hand, kurtosis measures the 'heaviness' of the residual distribution's tails relative to a normal distribution. High kurtosis values indicate a concentration of small errors, accompanied by occasional large deviations, which can disproportionately affect the RMSE. To further enhance the robustness of the analysis, we included the median absolute error (MedAE). Unlike RMSE and MAE, MedAE does not rely on assumptions about residual distribution and is more resistant to the influence of outliers, providing a reliable measure of the typical magnitude of errors in non-normal contexts.

$$MedianAE = median\ |SS_{residual}| \qquad (7)$$

Complementing the use of RMSE, p-values from the Shapiro–Wilk test were calculated to assess residual normality. For MAE, we applied the Kolmogorov–Smirnov test against a uniform distribution

(KS-uniform) to determine whether residuals deviated from uniformity. Both tests were implemented in the base R stats package [35].

2.3.4. Summarizing precision and accuracy

The concordance correlation coefficient (CCC) was used as a final summary of model performance in each of the sample subset scenarios. CCC was explicitly designed to assess the ability of one measurement method to reproduce the results from a 'gold-standard' method [36].

$$CCC = \frac{2\rho \ \sigma x \sigma y}{\sigma_x^2 + \sigma_y^2 + \left(\mu_x + \mu_y\right)^2} \qquad (8)$$

Where ρ is the Pearson correlation coefficient between the two measurements, σ_x^2 and σ_y^2 are the corresponding variances, μ_x and μ_y are the means of the two measurement methods. Accounting for both systematic bias and random variation simultaneously is useful in summary, but not necessarily diagnostic, because multiple factors could explain differences in CCC results [37]. Nonetheless, while not immune to differences in variance, the CCC is the most appropriate when the range and variance of the underlying real-world SOC concentrations are comparable.

The ability to predict DC-SOC from LOI measurements was tested under varying scenarios of LOI treatment and soil conditions, stemming from contrasting soil parent materials and horizons. While acknowledging the issues associated with comparing model performance statistics across different sample sets, we consider expected trends with increasing variance and examine the use-case impacts of the various components of accuracy and precision, as measured by the suite of statistics employed.

2.4. Experimental design

The relationship between DC and the different LOI methods was quantified using linear models with least squares regression. The precision of each LOI method was compared using the R^2 statistic. Then, the accuracy of the conversion equations associated with each LOI method was evaluated. For each model, the intercepts of the linear regression equations were tested for significant differences from zero. A p-value less than 0.05 determined if the null hypothesis of the intercept equaling zero should be rejected. A positive intercept indicated incomplete removal of SOM, while a negative intercept signified that processes like dehydration, dehydroxylation, or carbonate removal likely occurred [2].

Localized conversion equations were developed based on IC content and landform region, and then compared to investigate which of these two factors affected precision and accuracy. Evaluation of region and horizon served as proxies for differences in clay mineralogy, where differences in the precision and accuracy of conversion equations for the respective subsets were assumed to be driven by the material content of the samples. Climate and vegetation were not considered to be variable enough across the Iowa landform regions to be likely factors. Comparison between the performance of region-specific and generalized conversion equations (i.e., based on the complete data set) served as a test of

whether localized conversion equations could compensate for confounding factors (e.g., non-SOM mass loss).

To compare the measurement errors of the different methods (i.e., repeatability), we calculated the coefficient of variation (CV), defined as the ratio of the standard deviation to the mean of replicate measurements. For LOI, triplicate samples were analyzed by different operators to capture both analytical and operator-related variability. For DC measurements, CVs were derived from duplicate quality-control samples included in each analytical batch.

3. Results and discussion

3.1. Assessment of relationships between DC and different LOI methods

3.1.1. Which LOI method has the highest precision?

The regression analysis on all sample points demonstrated a reasonable relationship between LOI-SOM measurements and DC-SOC, with R^2 values of 0.67 and 0.65 when using LOI360 and LOI400, respectively. In contrast, when using LOI550, R^2 decreased to 0.47 (Fig. 4). This decline in precision suggested that variability in mass loss with LOI550 may not be suitable for a generalized conversion equation. The breakdown in the relationship between LOI-SOM at 550°C and DC-SOC was most likely due to greater mass losses associated with dehydroxylation or CO_2 volatilization of minerals at the higher temperature. The smectite, vermiculate, chlorite, illite, and kaolinite content in the soil samples was susceptible to some mass loss at this higher temperature. Kaolinite could be especially impactful on the mass lost, with a mass loss of up to 14% (Table 1). Such impacts are difficult to predict quantitatively without prior quantification of those soil minerals.

3.1.2. Which conversion equations provide the highest accuracy?

The lowest MAE and RMSE values were obtained for LOI360 and LOI400, indicating smaller mean and extreme deviations between predicted LOI-SOC and observed DC-SOC values. In contrast, LOI550 produced the largest residuals (Fig. 5). The MedAE values were consistently smaller than RMSE and MAE, underscoring that most predictions were close to the observed values and that the larger deviations penalized by RMSE occurred only occasionally. The slight differences between RMSE, MAE, and MedAE for LOI360 and LOI400 suggest comparable predictive accuracy between these two treatments, whereas LOI550 displayed both larger typical and extreme errors (Fig. 5).

The residual diagnostics revealed substantial deviations from normality (Shapiro-Wilk $p < 0.001$), with heavy tails (kurtosis ranging from 6 to 8) and near-zero skewness for all LOI treatments (Supplemental Table S1). Under these non-Gaussian, heavy-tailed errors, MedAE provides more robust summaries of

the error. The slight differences in RMSE, MAE, and MedAE between LOI360 and LOI400 suggest comparable predictive accuracy, whereas LOI550 exhibits larger typical and extreme errors.

This behavior is consistent with additional mass losses due to dehydroxylation of minerals such as goethite, smectite, illite, and particularly kaolinite. Without knowledge of the concentrations of these potential additional sources of mass loss, the accuracy of conversion equations at higher temperatures of ignition weakens. CCC, as a summary statistic for precision and accuracy, reflects this same pattern with moderately good agreement for LOI360 and LOI400, and a decline for LOI550 (Fig. 5).

3.2. Examination of variability and sources of error

3.2.1. Effect of excluding IC

Comparison of models for samples without IC to those for the entire data set showed a simultaneous improvement in both precision and accuracy. For this subset, R^2 increased by 0.11 and 0.16 units over the full set regression for LOI360 and LOI400, respectively, despite the decrease in variance. At the same time, RMSE dropped by 0.13 (LOI360) and 0.17 (LOI400), MAE dropped by 0.06 (LOI360) and 0.09 (LOI400), while MedAE showed minor improvements for both LOI360 and LOI400 (Fig. 6). Although none of the error statistics are large, the pattern of higher error for samples with IC suggests that either IC or something correlated with IC reduced the precision and accuracy of the conversion equations.

The most substantial effect of not including samples with IC was observed in precision (Fig. 6). The R^2 for LOI550 was relatively low with or without IC, but the difference in R^2 between with and without IC for LOI360 and LOI400 was large. LOI360 and LOI400 were highly precise ($R^2 > 0.78$) for samples without IC; however, the R^2 values dropped below 0.54 for these methods on samples with IC. The variance of SOC for samples with IC (0.80) is only slightly less than that for samples without IC (0.88), which makes the difference in variance an unlikely explanation for the large reduction in R^2.

The exclusion of IC samples resulted in some improvement in the accuracy of the LOI-SOC predictions of DC-SOC (Fig. 6C–D), although not as pronounced as the difference in precision. This result suggests that the conversion equations compensate well for the additional sources of mass loss. Residual diagnostics continued to show substantial deviations from normality in all cases (Shapiro–Wilk $p < 0.001$) and heavy-tailed residual distributions (kurtosis 5–9), with near-zero or mild skewness (Supplemental Table S1). However, the exclusion of samples with IC reduced both kurtosis and error magnitudes. Nonetheless, the gain in accuracy was never more than 0.22% SOC in terms of RMSE, and even less for MAE and MedAE.

Lower CCC for models calibrated on samples with IC reflects the tendency to underestimate SOC at higher concentrations. Models trained on samples that experience mass loss beyond SOM tend to underestimate SOC. Although the effect is small, it may be enough to reduce the determination of statistical significance when the SOC change is also small.

To understand the drivers of reduced model performance, it is essential to consider the known interactions between soil materials and temperature. None of the forms of carbonate are known to volatilize CO_2 below 425°C, making the presence of IC an unlikely direct cause of reduced precision for LOI360 and LOI400. However, given the observed relationship between IC and the reduced performance of LOI to predict SOC, IC is an indicator of likely lower precision and accuracy.

3.2.2. Effect of stratification by region

Comparing precision between regions and the data set as a whole becomes problematic due to the range of variances for those sample sets (Table 3). However, the trend of decreasing variance does not fully account for the differences in R^2 between regions (Fig. 7). The conversion equations for DML and IES exhibited precision comparable to that of the overall data set. The R^2 for the SIDP was higher and for the NWP lower than expected. For the SIDP, the conversion equations appear to compensate well for a consistent excess of mass loss with increasing LOI temperature. The y-intercept decreases from − 0.35 to -1.5 when transitioning from LOI360 to LOI550, but across all three methods, the R^2 remains high. For the NWP, the low R^2 values indicate low precision that is likely driven by greater variability in soil components contributing to mass loss beyond SOM. Here, that higher variability in mass loss is more noticeable with the lower variance. These results suggest that localized conversion equations can be beneficial if the additional sources of mass loss are consistent; however, they can be challenging if the causes of these additional mass losses are variable and not measured.

Table 3
SOC descriptive statistics for all samples compared to regional sample sets. Although not the case for the combined data set, variance decreased as the quantity of samples decreased.

	All	DML	IES	NWP	SIDP
n	423	168	134	92	29
Mean	1.02	0.97	1.27	0.81	0.71
Variance	0.96	1.4	0.71	0.6	0.35
Min	0.00	0.00	0.00	0.00	0.16
Max	7.58	7.58	4.93	3.56	2.08

Although the local models reduced error compared to the general model, the magnitude of these improvements remained modest. The maximum reductions were 0.46%, 0.32%, and 0.24% SOM for RMSE, MAE, and MedAE, respectively. These values are all below 0.5 percentage units of SOC, which can be considered the approximate threshold at which differences begin to acquire practical significance. These reductions are relatively small in operational terms. Nevertheless, even minor improvements in accuracy may influence the detection of SOC over time, and the implications of these reduced errors for detecting changes in SOC over time are examined further in Section 3.3.

While differences in variance make comparing precision between sample sets unreliable, the metrics of accuracy indicate little benefit for creating localized conversion equations by region. The summary statistic CCC suggests some improvements with the localized conversion equations for SIDP and DML. However, except for LOI550, the maximum increase in CCC was 0.15. That increase in CCC, which was for the smaller SIDP subset of 29 samples, may not be sustained with an increase in sample quantity.

Whatever the differences in mineralogy were among the soils in different landform regions of this study, they were not sufficient to dramatically alter the precision or accuracy of converting LOI-SOM measurements to DC-SOC. While the SIDP samples benefited from a localized conversion equation, the similar NWP samples did not. Because soil parent materials in the SIDP and NWP regions both contain smectite and illite, the difference in model performance between these two regions is unlikely to be explained by their clay mineralogies alone. This analysis suggests that the variability of clay mineral concentrations is a more significant factor in localized conversion equations than the type of clay minerals alone. That is, if the relative contributions of clay minerals to mass loss are consistent, then a conversion equation can compensate. Although precision and accuracy appear to improve with localization for the SIDP, this improvement may be offset if additional samples reveal greater clay mineral variability in the region.

3.2.3. Generalized versus localized conversion equations

In summarizing this analysis, the focus is placed on CCC, a metric designed for comparing two methods of measurement, and the metrics of prediction accuracy, as they are most closely connected to the practical implications of measurement error. Across the three LOI methods, localizing the conversion equation for LOI-SOM to DC-SOC showed some improvement in model performance nearly half of the time (Figs. 8 and 9). However, there were more improvements for the methods that tended to either underestimate (i.e., LOI360) or overestimate (i.e., LOI550) SOM. This pattern suggests that local models are compensating for the interactions between methods and local soil characteristics. For example, the most improvements occurred for LOI550, where high temperatures interact with varying concentrations of clay minerals that are susceptible to mass loss.

The segmentation of the data set into finer and finer local conditions highlighted the challenges of obtaining sufficient quantities of samples for increasingly specific models for conversion. Many localized models that exceeded the generalized model's performance (red dashed line) tended to have a small sample size ($n < 10$), which can lead to an overfit model that would decline in performance if more samples were analyzed. This interpretation is reinforced by the fact that in many cases, where CCC increases, MedAE does not decrease proportionally.

The contrast between the performance of localized models for SIDP and NWP illustrates the interaction between sample set size, CCC, and MedAE. The SIDP showed improvements in CCC for the majority of the data segmentations, but the entire region had only 29 samples, and subdivisions of those samples were even fewer. In contrast, the NWP showed minimal to no improvements in CCC from localization, as only two of the segmentation sets had fewer than 20 samples. Although the MedAE decreased below

the MedAE for the generalized model in most of the SIDP's localized models, the difference was a maximum of 0.1% SOC, excluding localized models with fewer than 5 samples and a variance of 0.01 or less. While the generalized models are broadly applicable across the presence of IC, landform regions, and horizons, localized models should be used only when the number of samples is large, and there is confidence that the sample variance represents the population.

The overall results indicate that localized calibrations rarely outperform the generalized models in a substantial or robust manner. Because of this, we can assume that the generalized equations remain broadly applicable, and specialized models from narrowly focused sample sets should be used only in cases where an adequately large sample size and consistent reductions in error metrics are observed. If other factors for mass loss (i.e., clay mineralogy) can be accounted for, then the precision and accuracy of predicting DC-SOC from LOI-SOM could be improved. However, while landform regions, horizons, and the presence of IC showed some correspondence with these confounding factors, they were not sufficient proxies for yielding strong improvements.

3.3. Evaluation of measurement error

Precision in the form of consistent measurements influences the accuracy of calculating differences between samples. Repeated measurement of samples demonstrated DC as the method with the highest precision by having the lowest CV. Among the LOI treatments, LOI400 had the highest precision with a CV of 5.3%, followed by LOI360 with 7.6% and LOI550 with 26.9% (Table 4). Although the different LOI treatments had lower precision than DC, LOI400 and LOI360 still had suitable levels of precision for many applications. These results are consistent with [24], who measured SOC using DC and SOM using LOI360. They reported a CV of 2.6% for DC-SOC, identical to our findings, and a CV of 4.1% for LOI360, which is 3.5 percentage points lower than our results. However, a higher CV in our study is expected, as we included samples from multiple landform regions, including soil with and without IC, and from A, B, and C horizons. In contrast, [24] evaluated samples from different landform regions separately and did not include any samples from C horizons.

Table 4
Descriptive statistics and CV as a measure of precision for DC and three treatments of LOI. DC-SOC had the highest precision, but lower temperature LOI methods were not substantially less precise.

	DC-SOC	LOI360-SOM	LOI400-SOM	LOI550-SOM
Mean (%)	1.70	2.08	2.88	4.25
Minimum (%)	0.24	0.10	0.35	1.33
Maximum (%)	4.90	5.41	6.71	7.10
n	73	45	45	45
CV (%)	2.60	7.60	5.30	26.90

n = number of samples, CV = coefficient of variation expressed in percentage.

Under emerging soil C market scenarios, uncertainty in SOC measurements is an important limitation that, for example, contributed to the collapse of the Chicago Climate Exchange voluntary carbon market [38]. Estimates of SOC stock accuracy are influenced by soil spatial variability, sampling design, and the laboratory method implemented [39]. Regarding the laboratory methods, they must be able to detect small changes under heterogeneous circumstances [40].

Some authors have argued that if the goal is measuring SOC, carbon should be measured directly using methods like DC (e.g., [2]). However, others have expressed concerns about the analytical variability of DC [5, 41]). The present study found that DC indeed offers superior replicability compared to LOI. However, LOI, particularly LOI400 and LOI360, also demonstrate satisfactory precision. For instance, based on the obtained CVs for SOC measurement, when analyzing a sample with 3% SOC, DC exhibited a variability of approximately ± 0.078%. In contrast, LOI400 and LOI360 presented a variability of ± 0.16% and 0.23%, respectively.

Although DC demonstrates the highest precision, accounting for spatial variability is crucial in detecting changes in SOC. Besides the equipment cost, cumulative analysis costs must also be considered, as higher spatial variability necessitates larger samples. For measuring SOC by DC, the price in a commercial laboratory in the Midwest USA is around $25, while the cost of SOM measurement using LOI is around $2. At a sampling rate of one sample per hectare [42], a 65-ha field would cost $1,625 using DC and $130 for LOI (Fig. 10). Conversely, if the goal were to stay within a budget or to maximize the spatial support for characterizing the SOC in that 65-ha field, $1,625 would provide either 65 samples of DC or 812 samples of LOI. While the CV for LOI400 may be double that of DC, the cost of LOI would enable the analysis of 12 times as many samples as DC.

Consider the relationship between the sample standard deviation (s) and the number of samples (n) in the paired t-test. The denominator for calculating t is s divided by the square root of n. If we assume that measurement error is the primary source of variability represented by s, then increases in s could be compensated for by increasing n. The s for the SOC samples analyzed by LOI400 (1.52%) was about three times that of the s for DC (0.442%). For LOI400 to have the same denominator in the t-test as DC does with 100 samples, 1,183 samples would be required. The costs become similar with these quantities of samples and the additional labor involved with collecting more samples. However, this exercise assumed that the variability in the samples came only from measurement error. Any real spatial variability in the samples collected would also increase s, which means that the more SOC varies in a field, the more the DC scenario would be at a disadvantage to the LOI400 scenario. For example, if field variability added one unit of standard deviation to the current scenarios, the denominator for the t-test of DC would be double that of LOI400, halving the t-value and greatly increasing the threshold for statistically significant differences with the t-test.

Based on these results, certifiers now have the necessary information to make an informed decision. DC is a method with high precision but a higher cost. The expense of DC limits the number of samples

supporting statistical significance tests, which is especially critical under high spatial variability conditions. In contrast, LOI400 has a slightly lower precision but a substantially lower cost. By only sacrificing measurement uncertainty to an RMSE of 0.69 g kg^{-1}, the cost savings can be invested in collecting more samples to enable better assessment of spatial variation.

Together with the lower cost and, consequently, better assessment of spatial variation, an additional benefit of collecting a higher number of samples when performing LOI could greatly facilitate the generation of expanded SOC training data sets that support emerging remote sensing and machine learning approaches for SOC estimations. Modelers' SOC prediction frameworks increasingly rely on algorithms calibrated with ground samples to capture spatial variability across contrasting landscapes. Because LOI is inexpensive, fast, and with good repeatability, it would enable researchers to assemble much denser calibration sample sets that would be economically feasible using DC alone. Having larger sample sets could be particularly valuable for training multi-sensor SOC models that utilize different indices (e.g., NDVI, SAVE, EVI, etc.), topographic attributes, and other covariates to create robust prediction models for mapping SOC and evaluating its variation in space and time.

4. Conclusions

This research provides a comprehensive evaluation of three loss-on-ignition (LOI) methods for estimating soil organic carbon (SOC) and compares them to the standard dry combustion (DC) technique across diverse soil landscapes. The results demonstrate that LOI400 (400°C for 16 hours) stands out as a reliable alternative to DC for estimating SOC. While DC remains the benchmark for analytical precision, its high cost and operational complexity restrict the number of samples that can be feasibly analyzed, which is a critical limitation when addressing spatial variability in SOC stocks. In addition, SOM has meaning for other environmental and agronomic management questions, which increases incentives to monitor it beyond carbon markets.

LOI400 and LOI360 (360°C for 2 hours) both deliver comparable precision and accuracy for predicting DC-SOC, with LOI400 exhibiting the highest repeatability among the LOI treatments. In contrast, LOI550 (550°C for 3 hours) is less suitable for generalized SOC estimation due to increased measurement error resulting from mineral mass loss, particularly in soils with variable clay minerals, such as kaolinite, smectite, and illite. Although those additional sources of mass loss could be measured and compensated for in the conversion equation, such additional analysis would defeat the purpose of keeping costs lower than those of DC.

The conversion equations evaluated in this research are not intended to estimate the concentration of C in SOM. They are the relationships between the methods of LOI and DC, which mathematically compensate for additional sources of mass loss in soil samples. A critical question is whether a generalized conversion equation is sufficient or if localized equations are needed. That question is difficult to access due to the inherent differences in variance between sample sets. Nonetheless, this research scrutinized conversion equations based on practical strategies for localization, using samples

with varying textures, clay mineral abundance, and SOM concentrations. Evidence for only minor improvements was found. The accuracy of estimating DC-SOC from LOI400 and LOI360 was high, with only minor opportunities for improvement.

Excluding samples with inorganic carbon (IC) from the analysis substantially improves the precision of LOI-based SOC predictions, but not necessarily the accuracy. The development of localized conversion equations, tailored to specific regions or soil horizons, provides additional benefits only in areas with consistent clay mineral content. For most practical applications, generalized conversion equations are sufficient, simplifying SOC monitoring protocols and reducing the need for extensive mineralogical characterization.

A key advantage of LOI methods is their low cost, which enables much higher sampling densities. This increased sampling capacity can offset the slightly higher measurement and prediction errors compared to DC, allowing for more robust detection of spatial patterns and changes in SOC stocks. In scenarios where resources are limited or spatial variability is high, LOI400 provides a practical solution for assessing large areas for SOC and verifying carbon markets.

In summary, the choice between DC and LOI methods should be guided by the specific goals of SOC monitoring, available resources, and the degree of spatial variability in the study area. LOI400 is recommended for most applications, providing a balance between reliability and cost to achieve a suitable sampling density. Localized conversion equations may be justified in regions with consistent mineralogical influences; however, generalized equations are generally adequate for broader use. These findings support the adoption of LOI400, per the USDA-NRCS Kellogg Soil Survey Laboratory Methods Manual (2022), as a practical and scalable approach for estimating SOC. The generalized equation for converting LOI400-SOM to the DC-SOC equivalent was LOI-SOC = 0.365 * LOI-SOM − 0.039.

Declarations

Funding

The USDA-NRCS Soil and Plant Division and USDA Hatch Project IOW05772 provided funding for this research.

Clinical trial: not applicable.

Ethics, Consent to Participate, and Consent to Publish declarations: not applicable.

Competing interests: The authors declare that they have no competing interest.

Data availability: The data generated and analyzed during the current study is available from the corresponding author upon reasonable request.

Author Contribution

Luis Bentancor led the study and was responsible for conceptualization, study design, data collection, data curation, formal analysis, and writing the manuscript. Bradely Miller made substantial contributions to the development of the study, data interpretation, and manuscript revision. Michael Thompson and Lee Burras reviewed the manuscript and provided critical comments that improved the scientific quality and clarity of the work. Peter O'Brien contributed to manuscript review and revision and provided technical expertise and support for laboratory analyses related to dry combustion measurements. All authors reviewed and approved the final manuscript.

Acknowledgement

The authors thank Dustin Ehret, Meyer Bohn, and Nicola Timbas for providing soil samples included in this analysis. Analytical Laboratory staff Tim Watts and Amy Morrow at the USDA-ARS National Laboratory for Agriculture and the Environment (Ames, Iowa) generously conducted the dry combustion analysis

Data Availability

The data generated and analyzed during the current study is available from the corresponding author upon reasonable request.

References

1. Jackson RB, Lajtha K, Crow SE, Hugelius G, Kramer MG, Piñeiro G. The ecology of soil carbon: pools, vulnerabilities, and biotic and abiotic controls. Annu Rev Ecol Evol Syst. 2017;48:419–45. https://doi.org/10.1146/annurev-ecolsys-112414-054234.
2. Pribyl DW. A critical review of the conventional SOC to SOM conversion factor. Geoderma Elsevier B V. 2010;156(3–4):75–83. https://doi.org/10.1016/j.geoderma.2010.02.003.
3. Van Bemmelen JM. Über die Bestimmung des Wassers, des Humus, des Schwefels, der in den colloïdalen Silikaten gebundenen Kieselsäure, des Mangans u. s. w. im Ackerboden. Landwirtschaftlichen Vers Stn 1890;37:279–90.
4. Watson RT, Noble IR, Bolin B, Ravindranath NH, Verardo DJ, Dokken DJ, editors. Land use, land-use change and forestry. Cambridge (UK): Cambridge University Press; 2000. p. 375.
5. Chatterjee A, Lal R, Wielopolski L, Martin MZ, Ebinger MH. Evaluation of different soil carbon determination methods. Crit Rev Plant Sci. 2009;28:164–78. https://doi.org/10.1080/07352680902776556.
6. Global Soil Laboratory Network (GLOSAN). Standard operating procedure for soil total carbon: Dumas dry combustion method (GLOSAN-SOP-03). Rome. IT); Food and Agriculture Organization of

the United Nations; 2019.

7. Johns TJ, Angove MJ, Wilkens S. Measuring soil organic carbon: which technique and where to from here? Soil Res. 2015;53:717–36. https://doi.org/10.1071/SR14339.
8. Hoogsteen MJJ, Lantinga EA, Bakker EJ, Groot JCJ, Tittonell PA. Estimating soil organic carbon through loss on ignition: effects of ignition conditions and structural water loss. Eur J Soil Sci. 2015;66:320–8. https://doi.org/10.1111/ejss.12224.
9. Schulte EE, Hopkins BG. Estimation of soil organic matter by weight loss-on-ignition. Soil organic matter: analysis and interpretation. Madison (WI): Soil Science Society of America; 1996. pp. 21–31.
10. De Vos B, Vandecasteele B, Deckers J, Muys B. Capability of loss-on-ignition as a predictor of total organic carbon in non-calcareous forest soils. Commun Soil Sci Plant Anal. 2005;36(19–20):2899–921. https://doi.org/10.1080/00103620500306080.
11. Wang X, Wang J, Zhang J. Comparisons of three methods for organic and inorganic carbon in calcareous soils of northwestern China. PLoS ONE. 2012;7. https://doi.org/10.1371/journal.pone.0044334.
12. United States Department of Agriculture (USDA). Soil Survey Field and Laboratory Methods Manual. Soil Survey Investigations Report No. 51. Version 2. Washington (DC): USDA; 2022.
13. Ball DF. Loss-on-ignition as an estimate of organic matter and organic carbon in non-calcareous soils. J Soil Sci. 1964;15(1):84–92. https://doi.org/10.1111/j.1365-2389.1964.tb00247.x.
14. Davies BE. Loss-on-ignition as an estimate of soil organic matter. Soil Sci Soc Am J. 1974;38(1):150–1. https://doi.org/10.2136/sssaj1974.03615995003800010046x.
15. Duval C. Inorganic thermogravimetric analysis. 2nd ed. Amsterdam: Elsevier Publishing Company; 1963. pp. 298–9.
16. Weliky K, Suess E, Ungerer CA, Muller PJ, Fischer K. Problems with accurate carbon measurements in marine sediments and particulate matter in seawater: a new approach. Limnol Oceanogr. 1983;28(6):1252–9. https://doi.org/10.4319/lo.1983.28.6.1252.
17. Barshad I. Thermal analysis techniques for mineral identification and mineralogical composition. In: Black CA, editor. Methods of Soil Analysis. Part 1: Physical andMineralogical Properties, Including Statistics of Measurement and Sampling. Madison (WI): American Society of Agronomy; 1965. pp. 699–742. https://doi.org/10.4324/9781315841205-9.
18. Christensen BT, Malmros PÅ. Loss-on-ignition and carbon content in a beech forest soil profile. Ecography. 1982;5(4):376–80.
19. Tan KH, Hajek BF, Barshad I. Thermal analysis techniques. In: Klute A, editor. Methods of Soil Analysis. Part 1: Physical and Mineralogical Methods. Madison (WI). Soil Science Society of America; 1986. pp. 151–83. https://doi.org/10.2136/sssabookser5.1.2ed.c7.
20. Karathanasis AD, Harris WG. Quantitative thermal analysis of soil materials. In: Dixon JB, Weed SB, editors. Quantitative Methods in Soil Mineralogy. Madison (WI): Soil Science Society of America; 1994. pp. 360–411. https://doi.org/10.2136/1994.quantitativemethods.c12

21. Jackson ML. Soil chemical analysis: advanced course. 2nd ed. New Delhi: United Book Prints; 1985. p. 930.
22. Sun H, Nelson M, Chen F, Husch J. Soil mineral structural water loss during loss on ignition analyses. Can J Soil Sci. 2009;89(5):603–10. https://doi.org/10.4141/CJSS09007.
23. Kohobhange SP, Karunadasa CH, Manoratne HM, Pitawala HMTGA, Rajapakse RMG. Thermal decomposition of calcium carbonate (calcite polymorph) as examined by in-situ high-temperature X-ray powder diffraction. J Phys Chem Solids. 2019;134:21–8. https://doi.org/10.1016/j.jpcs.2019.05.023.
24. Konen ME, Jacobs PM, Burras CL, Talaga BJ, Mason JA. Equations for predicting soil organic carbon using loss-on-ignition for north central U.S. soils. Soil Sci Soc Am J. 2002;66(6):1878–81. https://doi.org/10.2136/sssaj2002.1878.
25. U.S. Geological Survey. 3D Elevation Program (3DEP) digital elevation model (DEM) for the State of Iowa [raster dataset]. Reston (VA): U. S. Department of the Interior, U. S. Geological Survey; 2020. Available from: https://www.usgs.gov/3d-elevation-program
26. Handy RL, Lyon CA. Comparisons of petrographic and engineering properties of loess in southwest, east-central, and northeast Iowa. Proc Iowa Acad Sci. 1955;62(1):279–97.
27. Khan FAZM. Relationships of saturated zones and distribution of selected chemical and mineralogical properties in the Clarion catena [dissertation]. Ames (IA): Iowa State University; 1991.
28. Miller BA, Schaetzl RJ. Precision of soil particle size analysis using laser diffractometry. Soil Sci Soc Am J. 2012;76(5):1719–27. https://doi.org/10.2136/sssaj2011.0303.
29. Schepers JS, Francis DD, Thompson MT. Simultaneous determination of total C, total N, and 15N on soil and plant material. Commun Soil Sci Plant Anal. 1989;20(9–10):949–59. https://doi.org/10.1080/00103628909368128.
30. Sherrod LA, Dunn G, Peterson GA, Kolberg RL. Inorganic carbon analysis by modified pressure-calcimeter method. Soil Sci Soc Am J. 2002;66(1):299–305. https://doi.org/10.2136/sssaj2002.2990.
31. Chai T, Draxler RR. Root mean square error (RMSE) or mean absolute error (MAE)? Arguments against avoiding RMSE in the literature. Geosci Model Dev. 2014;7(3):1247–50. https://doi.org/10.5194/gmd-7-1247-2014.
32. Botchkarev A. A new typology design of performance metrics to measure errors in machine learning regression algorithms. Interdiscip J Inf Knowl Manag. 2019;14:45–76. https://doi.org/10.28945/4184.
33. Willmott CJ, Matsuura K. Advantages of the mean absolute error (MAE) over the root mean square error (RMSE) in assessing average model performance. Clim Res. 2005;30(1):79–82. https://doi.org/10.3354/cr030079.
34. Komsta L, Novomestky F. moments: Moments, cumulants, skewness, kurtosis and related tests. R package version 0.14.1. Vienna (AT): Comprenhensive R Archive Network (CRAN); 2022. Available from: https://cran.r-project.org/package=moments

35. R Development Core Team. R: A language and environment for statistical computing. Vienna (AT): R Foundation for Statistical Computing. 2025. Available from: https://www.r-project.org/
36. Lin LI. A concordance correlation coefficient to evaluate reproducibility. Biometrics. 1989;45:255–68. https://doi.org/10.2307/2532051.
37. Wadoux AMJC, Minasny B. Some limitations of the concordance correlation coefficient to characterise model accuracy. Ecol Inf. 2024;83:102820. https://doi.org/10.1016/j.ecoinf.2024.102820.
38. Gosnell H, Robinson-Maness N, Charnley S. Profiting from the sale of carbon offsets: a case study of the Trigg Ranch. Rangelands. 2011;33(5):25–9. http://dx.doi.org/10.2458/azu_rangelands_v33i5_gosnell2.
39. Stanley P, Spertus J, Chiartas J, Stark PB, Bowles T. Valid inferences about soil carbon in heterogeneous landscapes. Geoderma. 2023;430:116323. https://doi.org/10.1016/j.geoderma.2022.116323.
40. Lehmann J, Kinyangi J, Solomon D. Organic matter stabilization in soil microaggregates: implications from spatial heterogeneity of organic carbon contents and carbon forms. Biogeochemistry. 2007;85:45–57. https://doi.org/10.1007/s10533-007-9105-3.
41. O'Rourke SM, Holden NM. Optical sensing and chemometric analysis of soil organic carbon: a cost effective alternative to conventional laboratory methods? Soil Use Manag. 2011;27(2):143–55. https://doi.org/10.1111/j.1475-2743.2011.00337.x.
42. Mallarino A, Sawyer J. Take a good soil sample to help make good fertilization decisions [Internet]. Ames (IA): Iowa State University Extension and Outreach; 2023. Available from: https://store.extension.iastate.edu/product/3915

Figures

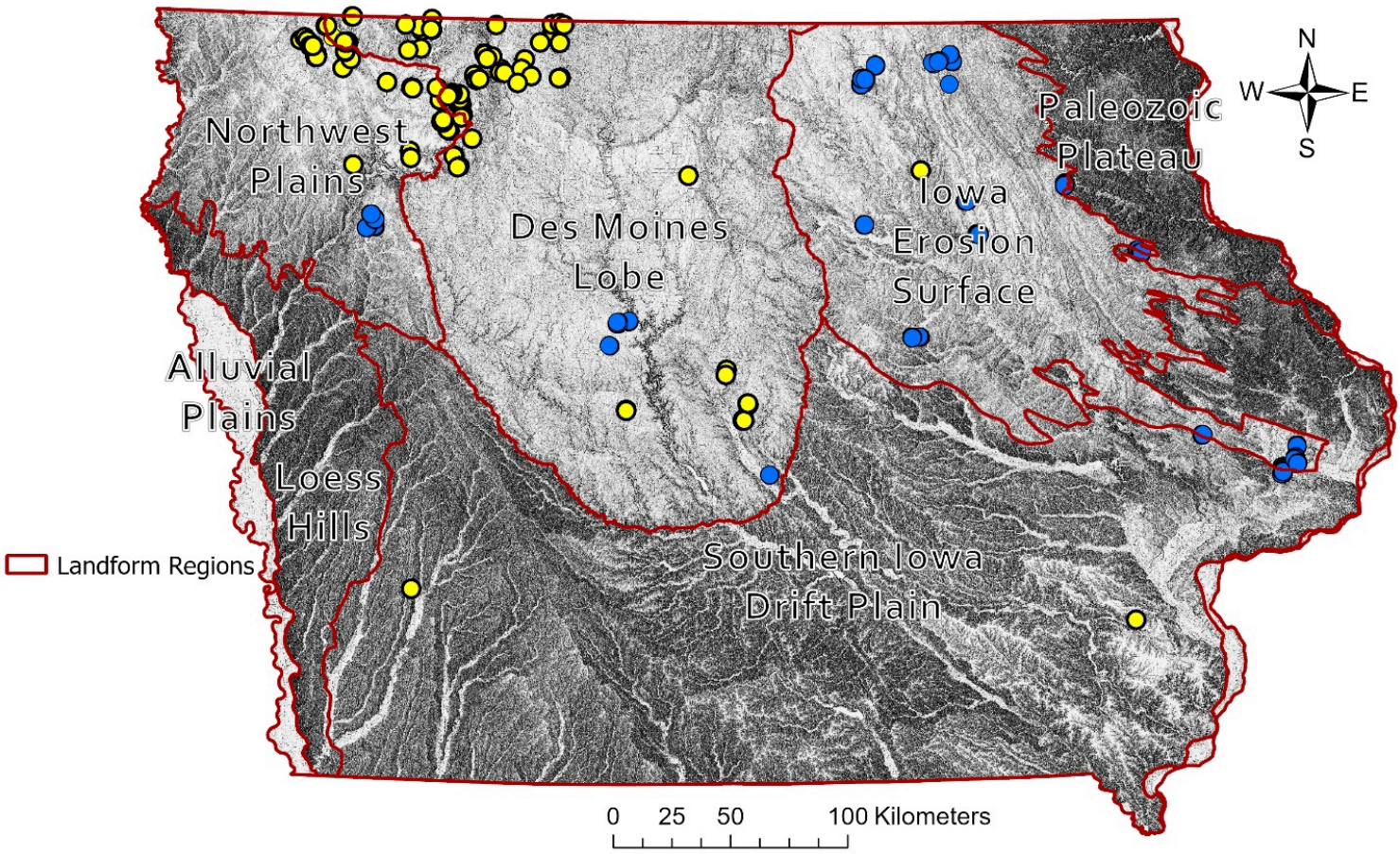


Figure 1

Distribution of sample locations within differing landform regions, shown on an elevation hillshade to indicate relief [25]. Blue locations were taken using a hand probe, while yellow locations were collected using a truck-mounted hydraulic soil probe (Giddings Machine Company, Windsor, CO).

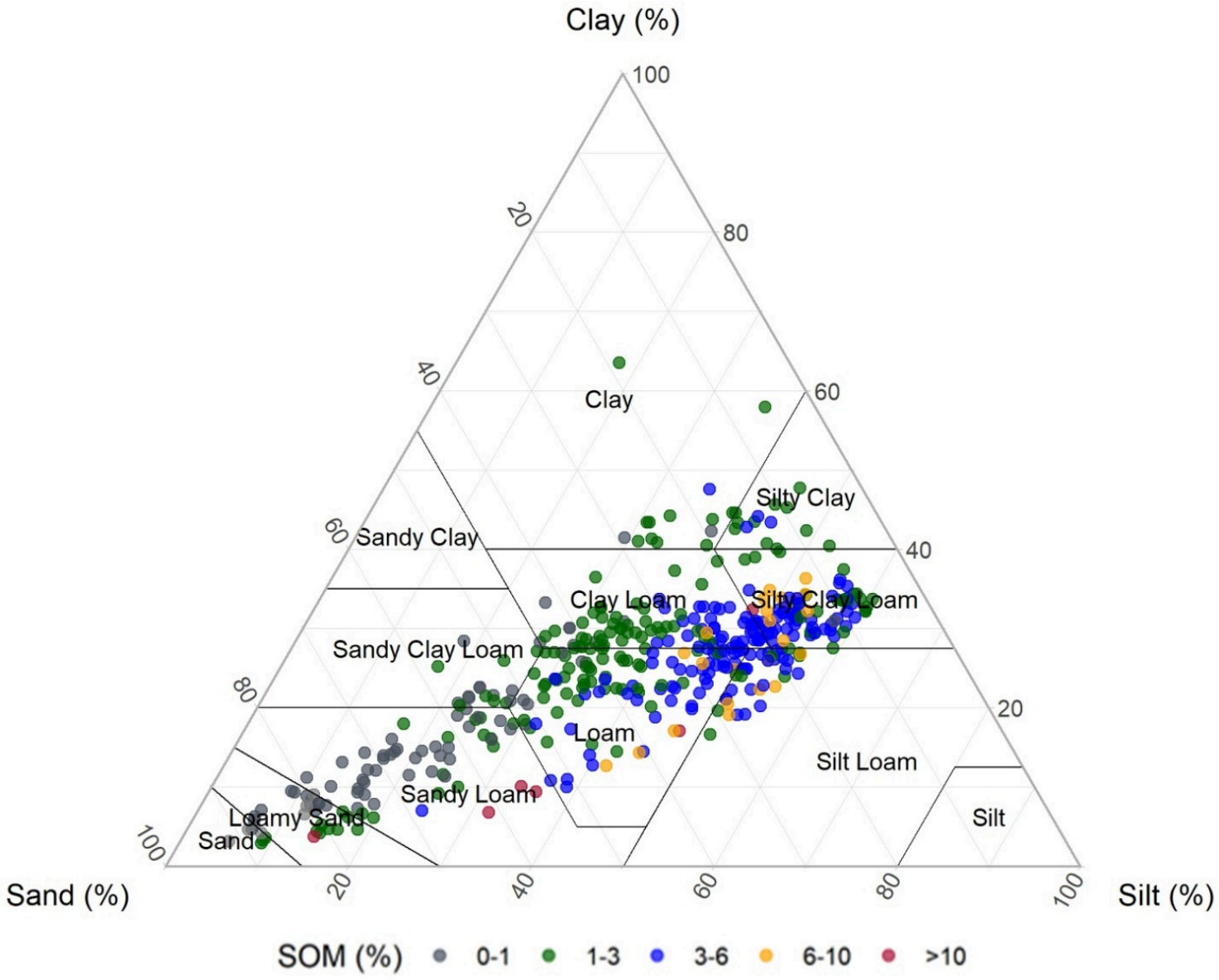


Figure 2

Distribution of soil texture class and SOM (%) concentrations of soil samples evaluated in this study. Different colors represent the range of SOM (%) concentrations.

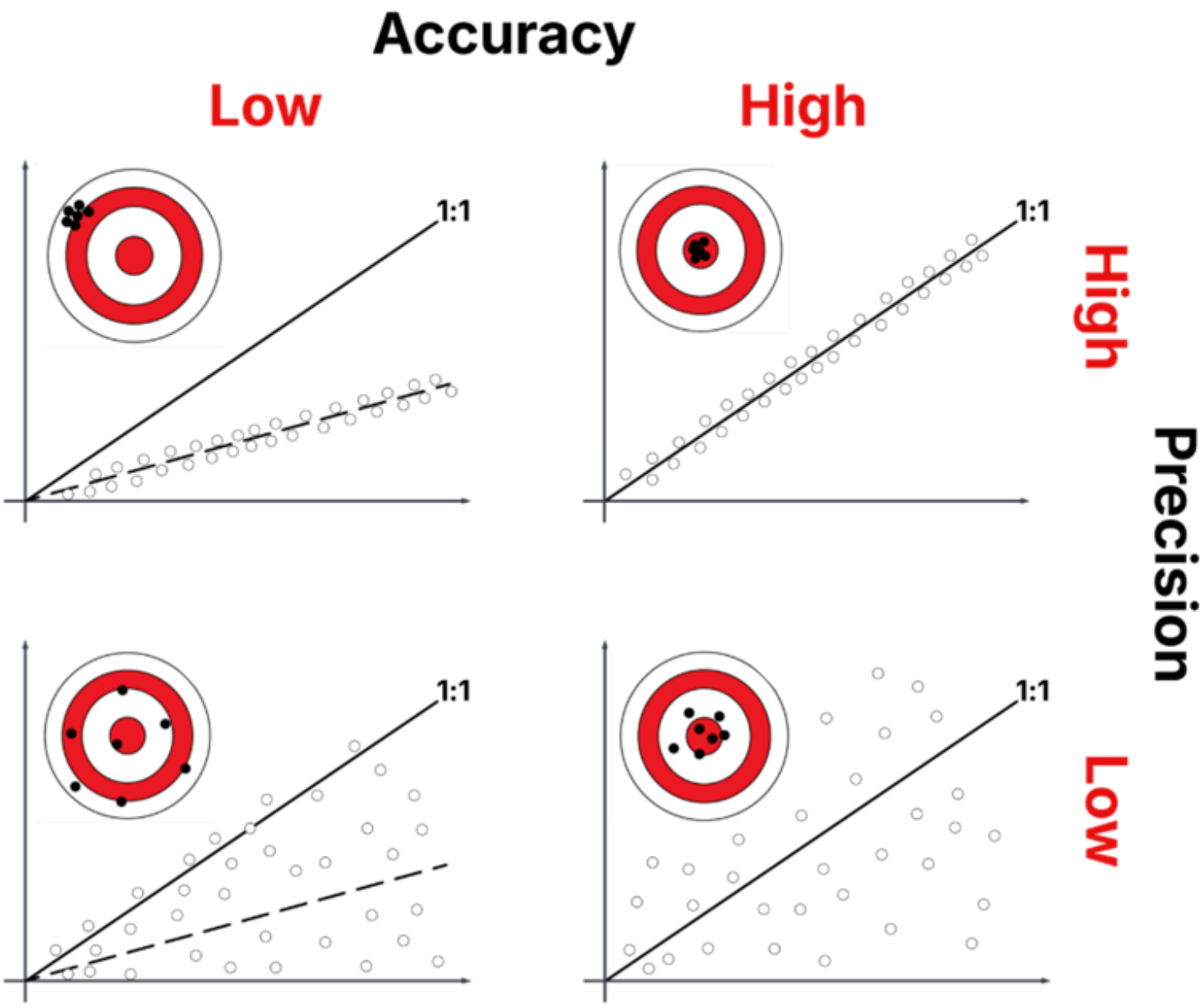


Figure 3

Conceptual illustrations of accuracy and precision, including the scatterplot scenario where the target is also a variable. The 1:1 line represents the perfect match between predicted values and observed values. The upper right panel demonstrates high accuracy and precision, with predictions closely matching observations and minimal scatter. The upper left panel shows low accuracy but high precision, where predictions are consistent but systematically biased. The lower right panel displays high accuracy but low precision, with predictions centered around the 1:1 line and exhibiting a large degree of scatter. The lower left panel shows low accuracy and precision, with predictions that are both biased and highly variable.

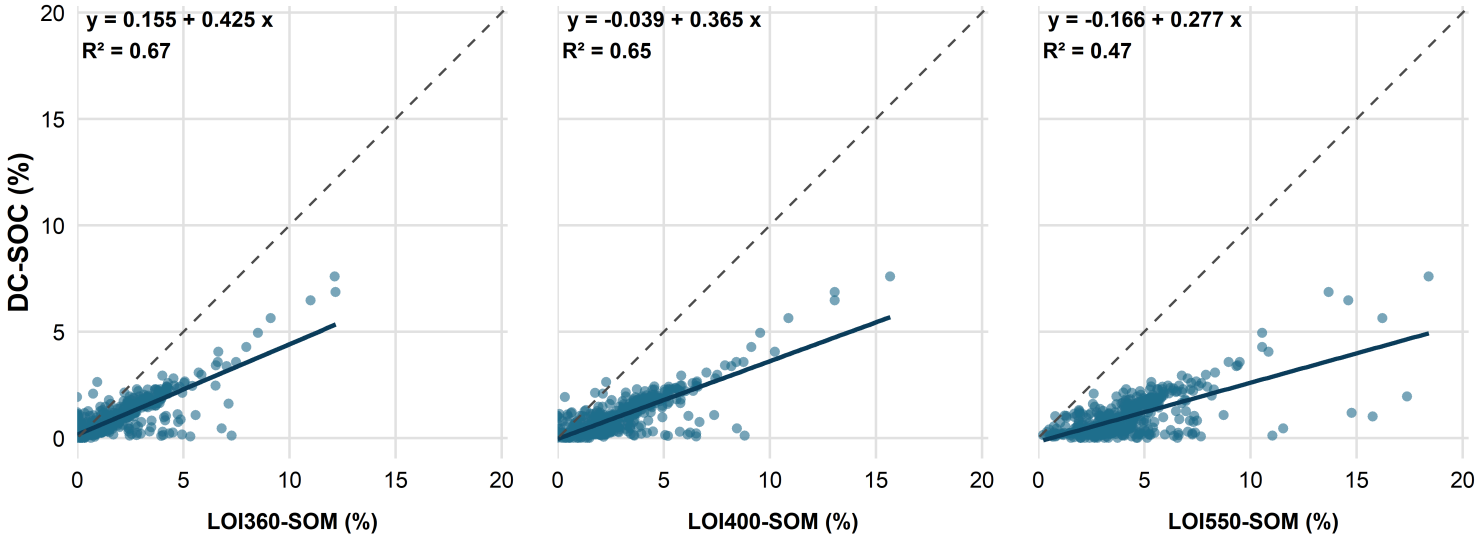


Figure 4

Relationship between LOI-SOM (%) and DC-SOC (%) for the three LOI treatments tested for the whole data set. The solid blue line shows the fitted linear regression, and the dashed line represents the 1:1 reference. The conversion equations shift from underestimating SOM mass for LOI360 to overestimating SOM mass for LOI400 and LOI550. The slope of the conversion equations decreases as the temperature of the LOI methods increases.

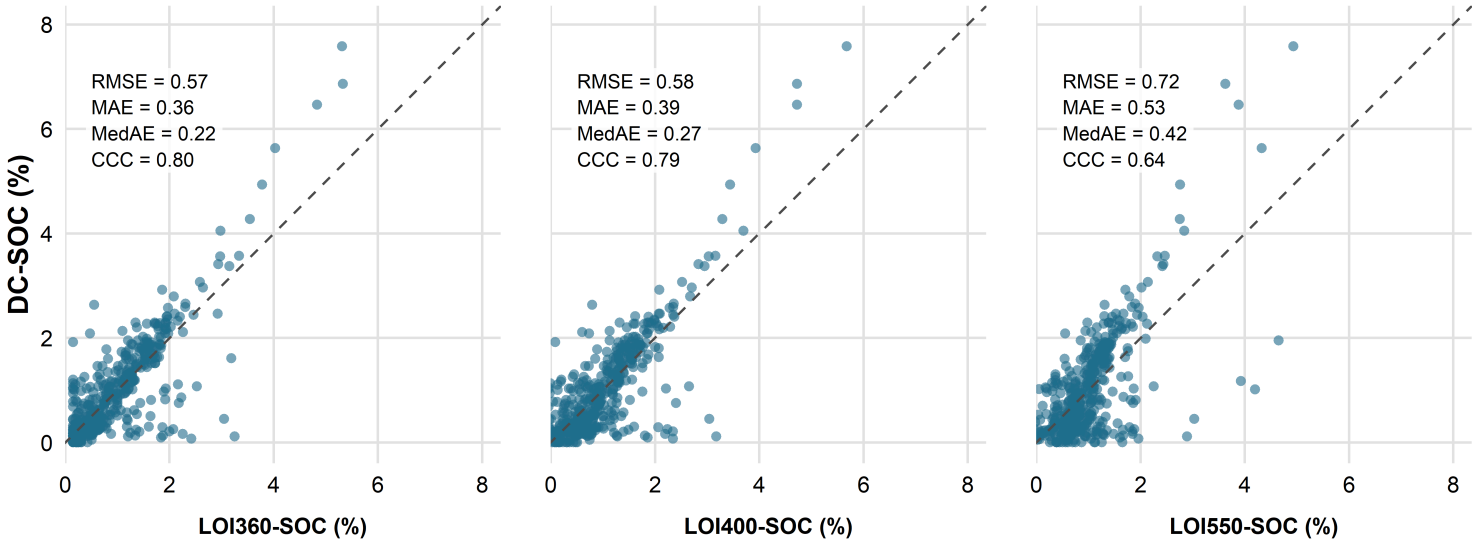


Figure 5

Performance of conversion equations based on the whole data set for the three LOI methods. The predicted LOI-SOC (%) is compared with the observed DC-SOC (%). Each panel shows the 1:1 reference (dashed line), where accurate predictions should align. RMSE, MAE, MedAE, and CCC are comparable for LOI360 and LOI400, while performance declines for LOI550.

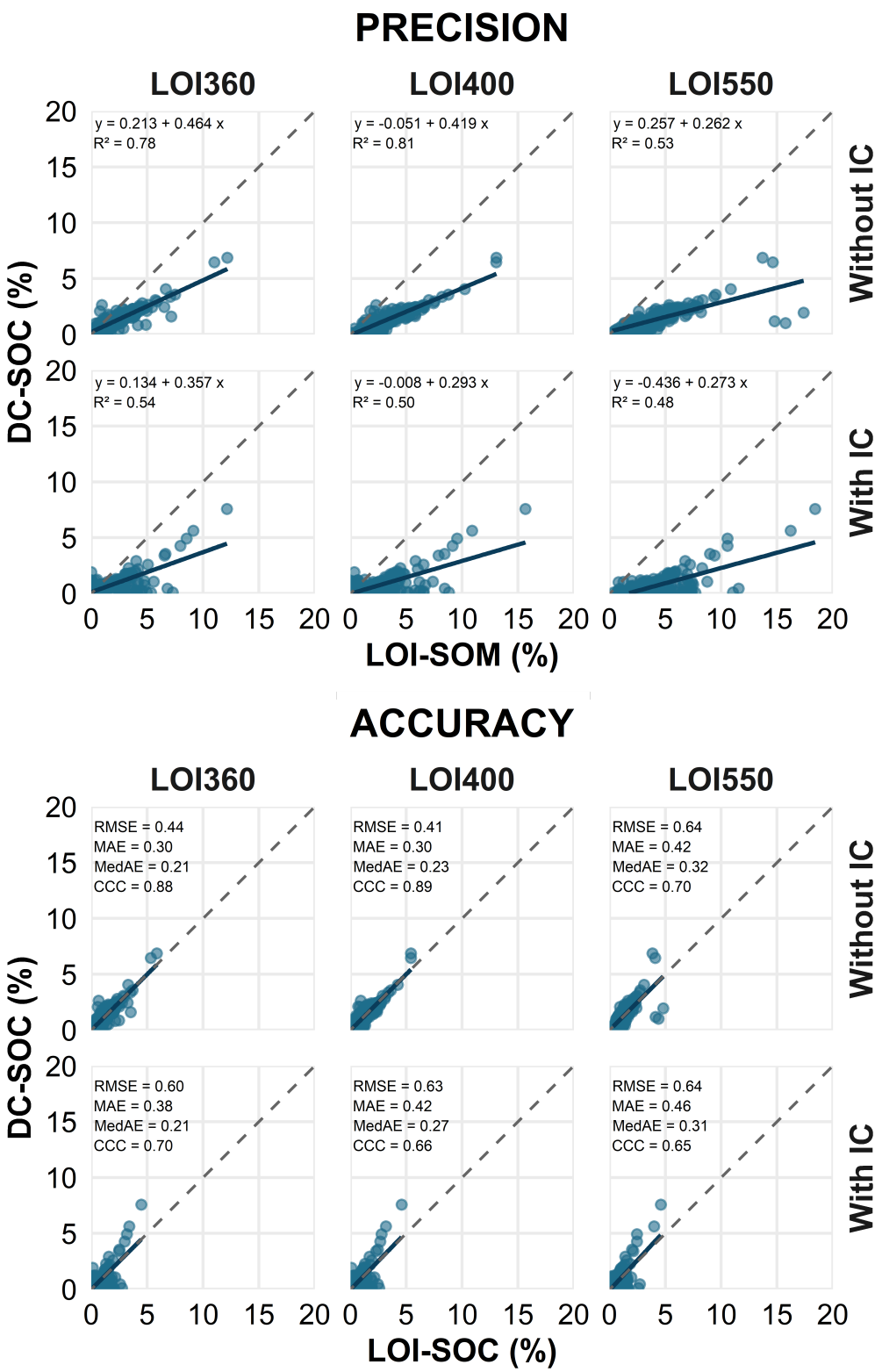


Figure 6

Comparison of the precision of the relationship between DC-SOC (%) and LOI-SOM (%), and accuracy of the predicted LOI-SOC and observed DC-SOC for samples with IC, and without IC. Essentially, samples without IC are less noisy.

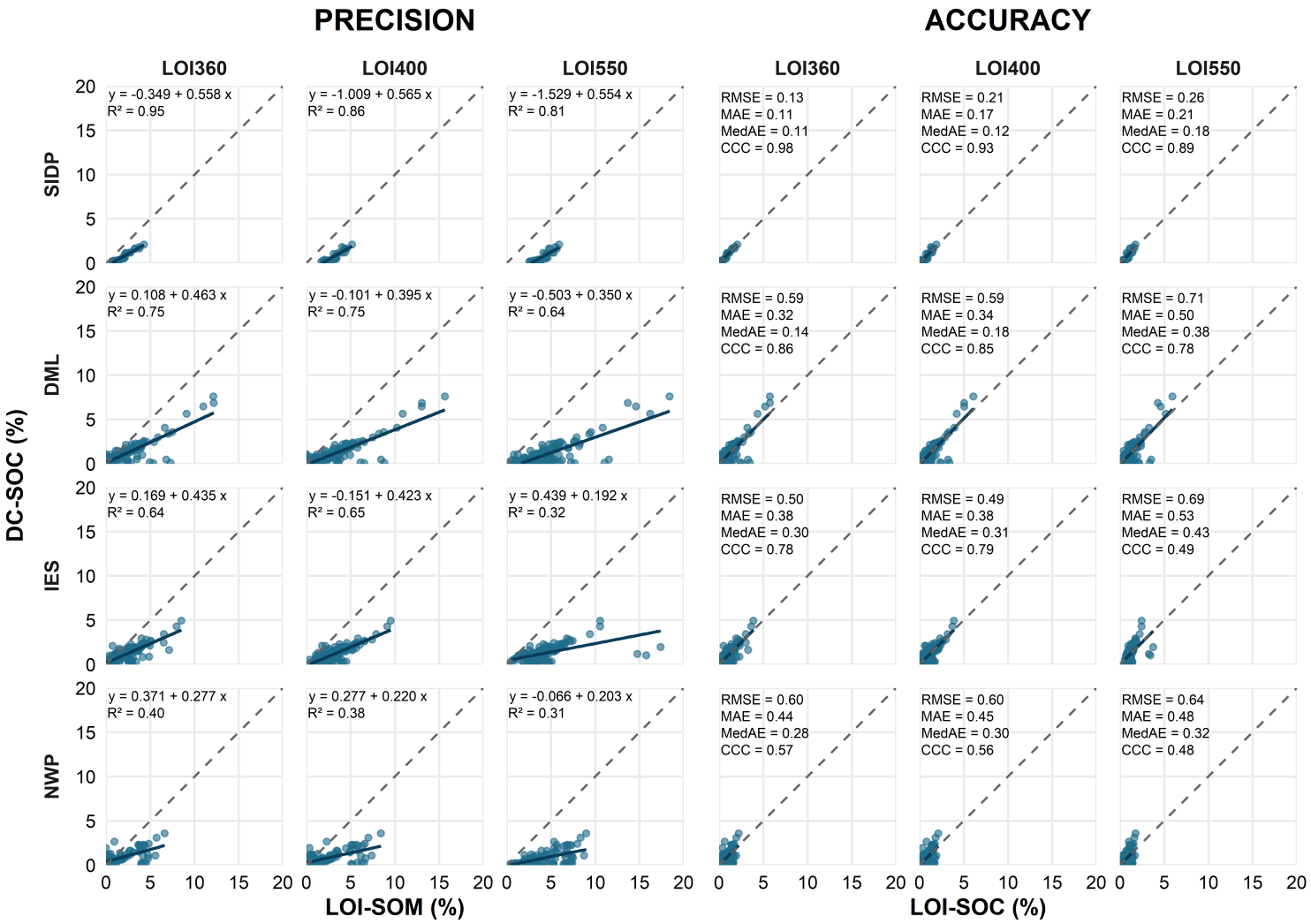


Figure 7

Comparison of the precision (left three columns) for the relationship between DC-SOC (%) and LOI-SOM (%) and the accuracy (right three columns) for the prediction of DC-SOC (%) by LOI-SOC (%) across the four landform regions (SIDP, DML, IES, and NWP) and three LOI methods. The results for the DML and IES generally align with those for the entire data set. The SIDP stands out with reduced variance and a corresponding increase in precision, as well as increased accuracy. The smaller number of samples for SIDP likely contributed to there being a more consistent amount of mass loss from non-SOM sources, which the conversion equation was able to accommodate better. The poor performance of the IES conversion equations across the board suggests the presence of more variable sources of mass loss in that localized sample set that are more difficult to account for in a simple conversion equation.

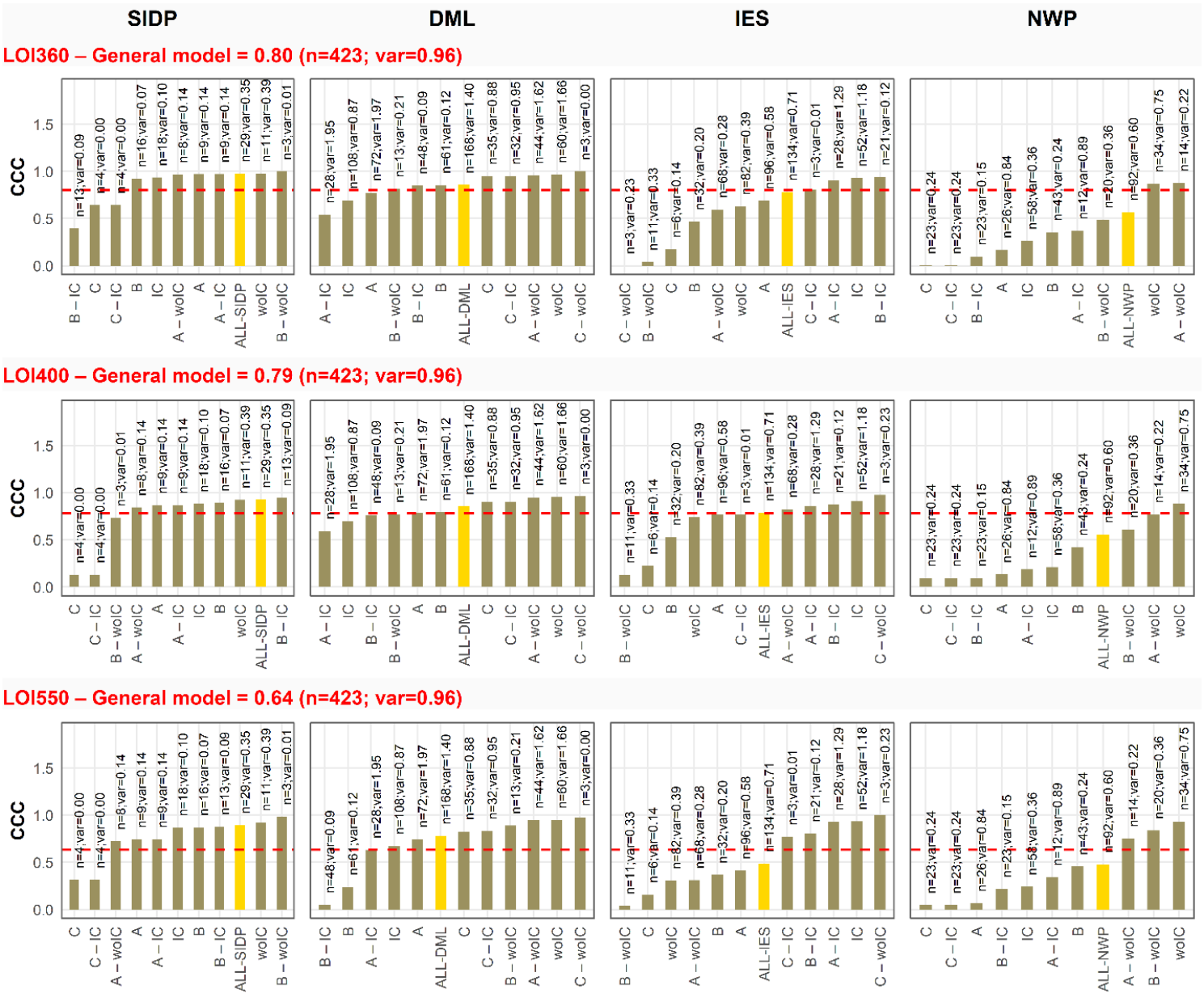


Figure 8

Comparison of CCC values between generalized and localized LOI-SOC models for LOI360, LOI400, and LOI550 across the four landform regions evaluated. The red dashed line represents the CCC of the generalized model using all samples (ALL) from this study, which serves as the reference for evaluation. Bars exceeding this line represent localized models with improved concordance relative to the generalized model. Yellow bars correspond to the models localized to the respective landform regions only (i.e., models built using all samples within each landform region), while olive bars represent models created by segmenting the data to more specific factors (IC content, horizon, or their combinations). Segmentations involving samples with IC are labeled “IC”, whereas segmentations involving samples without IC are labeled “woIC”. Horizon-based segmentations are denoted by A, B, and C. Although localized improvements are possible, the maximum improvement was a CCC change from 0.64 to 0.95, which occurred for the lowest-performing LOI550 method. Observed improvements greater than this are unlikely to be reliable due to the limited number of available samples.

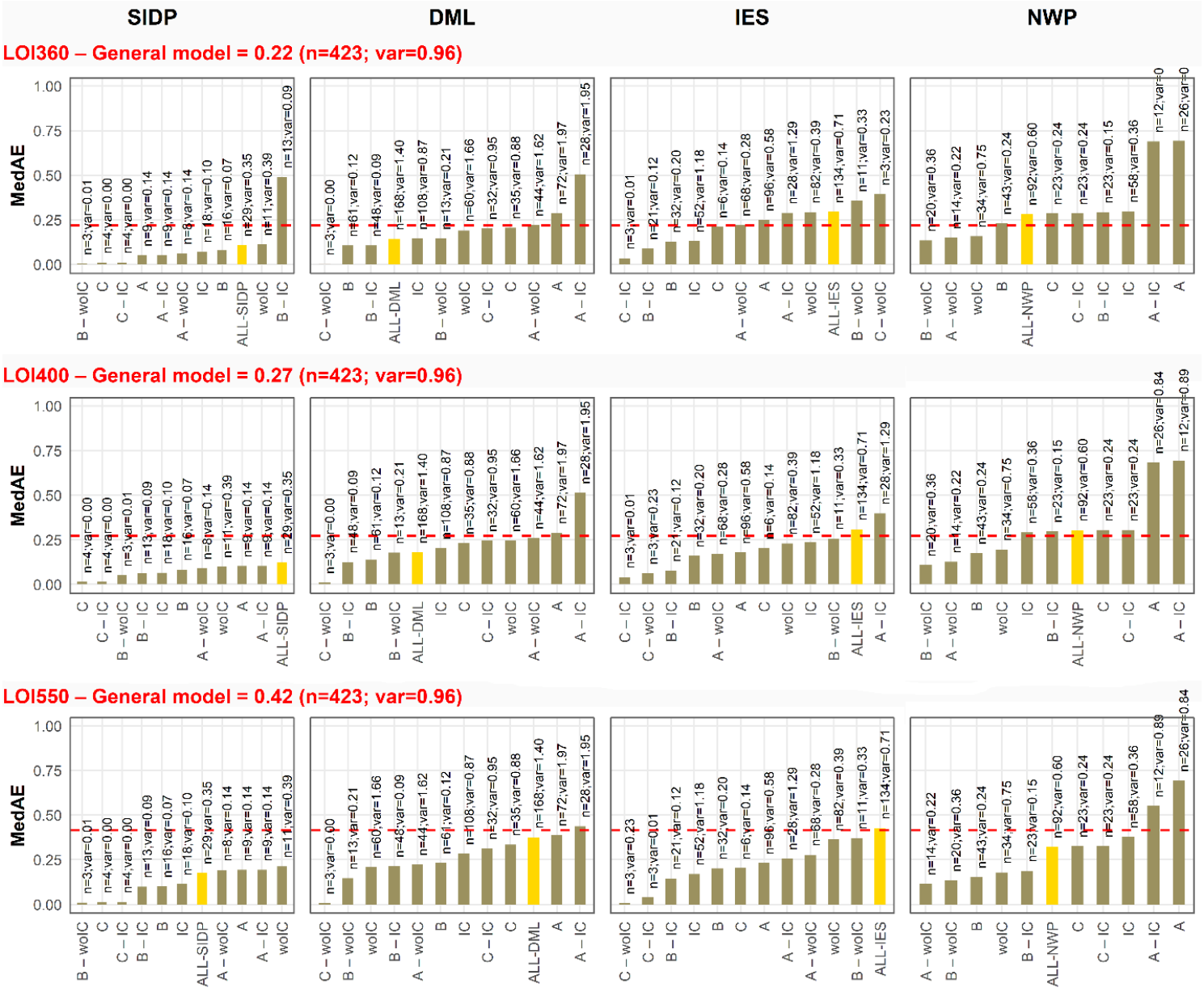


Figure 9

Comparison of MedAE values between generalized and localized LOI-SOC models for LOI360, LOI400, and LOI550 across the four landform regions evaluated. The red dashed line represents the MedAE of the generalized model using all samples (ALL) from this study, which serves as the reference for evaluation. Bars falling below this line represent segmented models with reduced error relative to the generalized model. Yellow bars correspond to the models localized to the respective landform regions only (i.e., models built using all samples within each landform region), while olive bars represent models created by segmenting the data to more specific factors (IC content, horizon, or their combinations). Segmentations involving samples with IC are labeled “IC”, whereas segmentations involving samples without IC are labeled “woIC”. Horizon-based segmentations are denoted by A, B, and C. Although localized improvements are possible, the maximum improvement was 0.37% SOC, which was for the least accurate LOI550 method. Observed improvements greater than this are unlikely to be reliable due to the limited number of available samples.

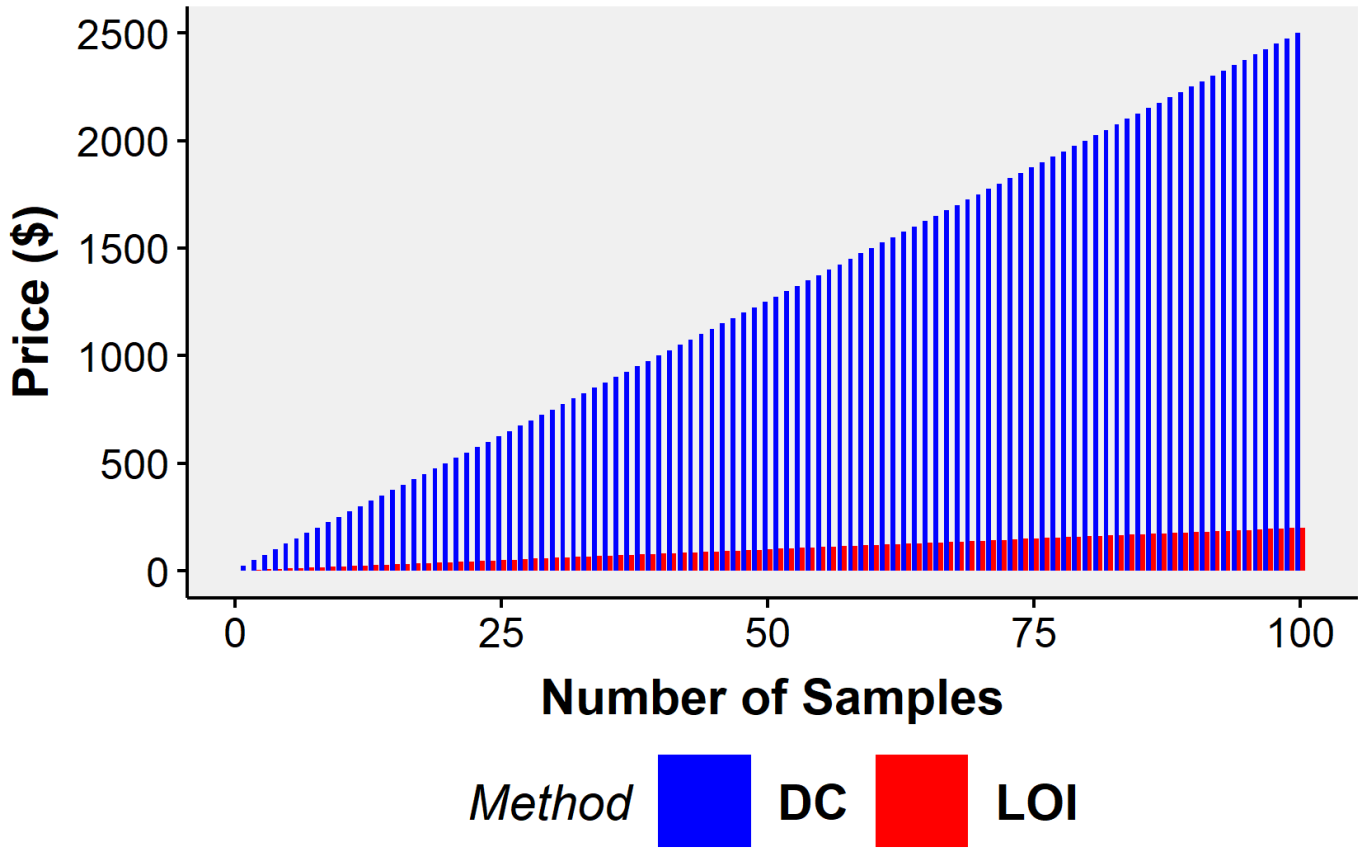


Figure 10

Price comparison by method. Prices for DC and LOI were based on the cost of a commercial lab in the USA Midwest, which was $25 and $2, respectively. This scale can be interpreted for either sampling larger areas or increasing sampling densities. With a focus on increasing statistical certainty for change detection, the lower precision of LOI can be compensated for by increasing the number of samples analyzed.

Supplementary Files

This is a list of supplementary files associated with this preprint. Click to download.

- SupplementalTableS1.docx