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Quantification of soil carbon and moisture content in varying-moisture soils with Laser-Induced Breakdown Spectroscopy and machine learning

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ABSTRACT

We combined laser-induced breakdown spectroscopy (LIBS) with LED-based vis–NIR reflectance and multivariate machine learning to predict total carbon (TC) concentration and gravimetric moisture content (MC) in variably processed soils under varying humidity. Using 116 samples from eight Finnish agricultural sites spanning six USDA texture classes, we defined five calibration scenarios from near-constant-humidity, ball-milled material to minimally processed samples with natural moisture. Six regressors were compared; kernel ridge regression performed best. On held-out test data from stratified 80/20 splits, TC prediction reached $R^2 = 0.95$ and MAE = 0.19 % TC for processed near-constant-humidity samples and $R^2 = 0.91$ and MAE = 0.30 % TC for minimally prepared fresh material, the most field-relevant case. MC was predicted with moderate accuracy on the fresh cohort alone ($R^2 = 0.70$) and excellent accuracy when all varying humidity states were pooled ($R^2 = 0.95$). Predicted moisture used as a feature significantly improved TC accuracy under natural varying moisture (MAE 0.36 vs. 0.30 % TC; paired *t*-test, $p = 0.005$). These results show that LIBS with auxiliary reflectance and machine learning can deliver useful TC and MC predictions from varying moisture soils with minimal sample preparation when moisture-aware calibration is applied.

Introduction

Soil is the second largest carbon storage on Earth, playing a central role in the global carbon cycle. Soil organic carbon (SOC) is predominantly concentrated near the soil surface making carbon storage highly sensitive to land use practices and land use change¹. In recent years, growing awareness of carbon emissions associated with primary food production combined with the expanding market interest in soil carbon sequestration as a carbon crediting mechanism have driven an increase in the demand for accurate, credible, and repeatable measurements of soil carbon stocks and their temporal changes². The spatial heterogeneity of SOC distribution in soils creates fundamental challenges for carbon stock quantification. The resulting uncertainty is primarily driven by insufficient spatial sampling density typically driven by high measurement costs^{2–4}. Closing this uncertainty gap requires analytical approaches capable of delivering high-throughput, spatially dense measurements at reduced cost.

Quantification of carbon stock requires an accurate measurement of carbon concentration and knowledge of the bulk density of the analyzed soil sample, making accurate carbon stock measurement even in an individual soil sample challenging. The bulk density can be determined by volumetric sampling, drying and weighing, whereas soil carbon concentration measurement requires a more sophisticated measurement approach. The quantification of carbon concentration relies on laboratory measurement methods, such as dry combustion⁵ and wet digestion⁶, which are laborious and time consuming, making carbon quantification expensive.

Optical spectroscopy offers a pathway towards fast, on-site soil analysis with acceptable measurement accuracy. By eliminating the need for sample transportation, storage, and labor-intensive pre-processing, field-deployable spectroscopic techniques would reduce per-sample analysis costs. Thus, optical soil carbon concentration measurement approaches can potentially enable greater spatial coverage with higher sample numbers, leading to more accurate soil carbon monitoring. Near-Infrared Reflectance Spectroscopy (NIRS) has been extensively applied to SOC analysis⁷. More recently, Mid-Infrared Reflectance Spectroscopy (MIRS) has gained considerable attention driven by the increasing commercial availability of compact mid-IR light sources and spectrometers⁸. Despite these advances, both NIRS and MIRS rely on empirical, correlation-based calibration models that require large, geographically representative reference datasets comprising typically thousands of laboratory-analyzed samples to achieve acceptable predictive accuracy^{9–11}. The transferability of such models beyond the

geographic and pedological domain used for their calibration remains limited, restricting the operational generalizability of these techniques⁸.

Laser-Induced Breakdown Spectroscopy (LIBS) is a fundamentally different approach to NIRS for on-site soil analysis. LIBS is a well-established elemental analysis technique¹². The technique is based on a pulsed laser that generates a laser-induced plasma with broadband atomic emission providing simultaneous, information-rich access to the elemental composition of the sampled material. This capability has been demonstrated across a wide range of industrial and environmental applications^{13–15}. In the context of soil science, LIBS has been explored for the detection and quantification of plant nutrients, heavy metals, and carbon¹⁶. However, the quantitative accuracy in soil analysis is complicated by matrix effects. Matrix effects are variations in signal intensity and relative peak ratios arising from the inherent heterogeneity of soil in terms of chemical composition, physical structure, and moisture content. These effects have been typically managed by extensive sample preparation, including drying, sieving, and grinding, which constrains the technique to laboratory or semi-laboratory settings and substantially undermines its on-site utility^{17–19}. Quantitative LIBS analysis of fresh, moist soils remains an underexplored and technically demanding challenge. Moisture introduces additional and highly variable matrix effects that are difficult to model with conventional calibration approaches.

In this work, we report on the quantitative determination of total soil carbon concentration in varying moisture soil samples using a combined LIBS and IR reflectance approach. In signal processing, we utilize the recent progress in Machine Learning (ML) that has redirected attention toward data-driven calibration models capable of capturing complex, non-linear spectral relationships²⁰. Neural Networks (NN), Support Vector Machines (SVM), and Random Forest (RF) classifiers and regressors have each been demonstrated in soil-related LIBS studies, covering applications from trace element quantification to soil classification and multi-parameter soil characterization^{21–23}. We evaluate the performance of these methods to determine the total carbon concentration and moisture content of soils under varying moisture conditions. Performance is compared across sample-processing schemes ranging from ball-milled material with constant-moisture to soils with minimal preparation. We also test whether the predicted moisture content, used as an additional feature, improves the accuracy of carbon prediction. The references for the carbon concentration and moisture content analyses were classical dry combustion (LECO 828, LECO Corporate, MI, US) and gravimetric moisture analysis, respectively.

Results

Laser-induced breakdown spectroscopy was conducted on a sample set of 116 soil samples with varying carbon and moisture contents. To compare sample processing practices, from each sample a portion was dried and ball-milled, leading into two duplicate sample sets with a difference in processing. A more detailed description of sample processing is presented in the Materials and Methods section. Spectra were collected with 2 spectrometers covering the range of ca. 190 nm - 950 nm. Figure 1 (a) demonstrates a single spectrum from a soil sample with a carbon concentration of 3.34 % in the UV range (190 nm - 450 nm). With modest (< 10 %) carbon concentrations, the most prominent carbon peak is often^{19,24} the peak from C I at 193.1 nm, as is the case in our sample set as well. The peak is highlighted with a grey shaded region in the plot. Fig. 1 (b) includes 5 other spectra divided with the spectra shown in the (a) panel of the same figure. These samples have almost exactly the same carbon concentration (3.28 % - 3.43 %) as the reference sample, but it is clearly evident from the figure that the spectra are highly divergent. Furthermore, panels (c) and (d) include the emission intensities (peak areas) of the C I lines of all samples as a function of carbon concentration. Panel (c) includes a subset of milled samples with a constant humidity, and panel (d) includes samples that have a varying humidity and are non-milled but have only the largest cm-size rocks removed. Clear correlation is seen with the carbon peak intensity, but even with the constant humidity case the prediction power using only the carbon peak is modest at maximum. With the varying moisture content, the prediction power would be poor.

Motivated by the results of Fig. 1, we constructed multiple multivariate models utilizing machine learning and neural networks to predict total carbon concentration (TC) (w/w) and moisture content (MC) (w_{H_2O}/w_{dry}) of the soil sample. The used models were Kernel Ridge (KR)²⁵, Support Vector Regression (SVR)²⁶, Partial Least Square regression (PLS)²⁷, Random Forest (RF)²⁸, Neural Network (NN)²⁹ and Extreme Gradient Boosting (XGB)³⁰. The same set of feature variables was used in both (carbon and moisture) predictions, and the details of the feature selection are presented in more detail in the Materials and Methods section. All samples were measured with three varying moisture contents and the ball-milled samples were also measured with constant humidity. The aim was to compare how sample processing affects model performance and to see if the effect of varying moisture illustrated in Fig. 1 (d) can be compensated by predicting moisture content before carbon concentration and using it as a feature in carbon prediction. Furthermore, we wanted to compare different modeling approaches to see which models perform best with our set of feature variables. The performance metrics of all experiments are shown in Supplementary Tables S1 and S2, which include the results of the total carbon measurements and the moisture content measurements, respectively. The hyperparameter search ranges and the results of the cross-validations for each model can be found in Supplementary Tables S4 and S5, respectively.

The data were divided into 5 groups depending on the sample preparation:

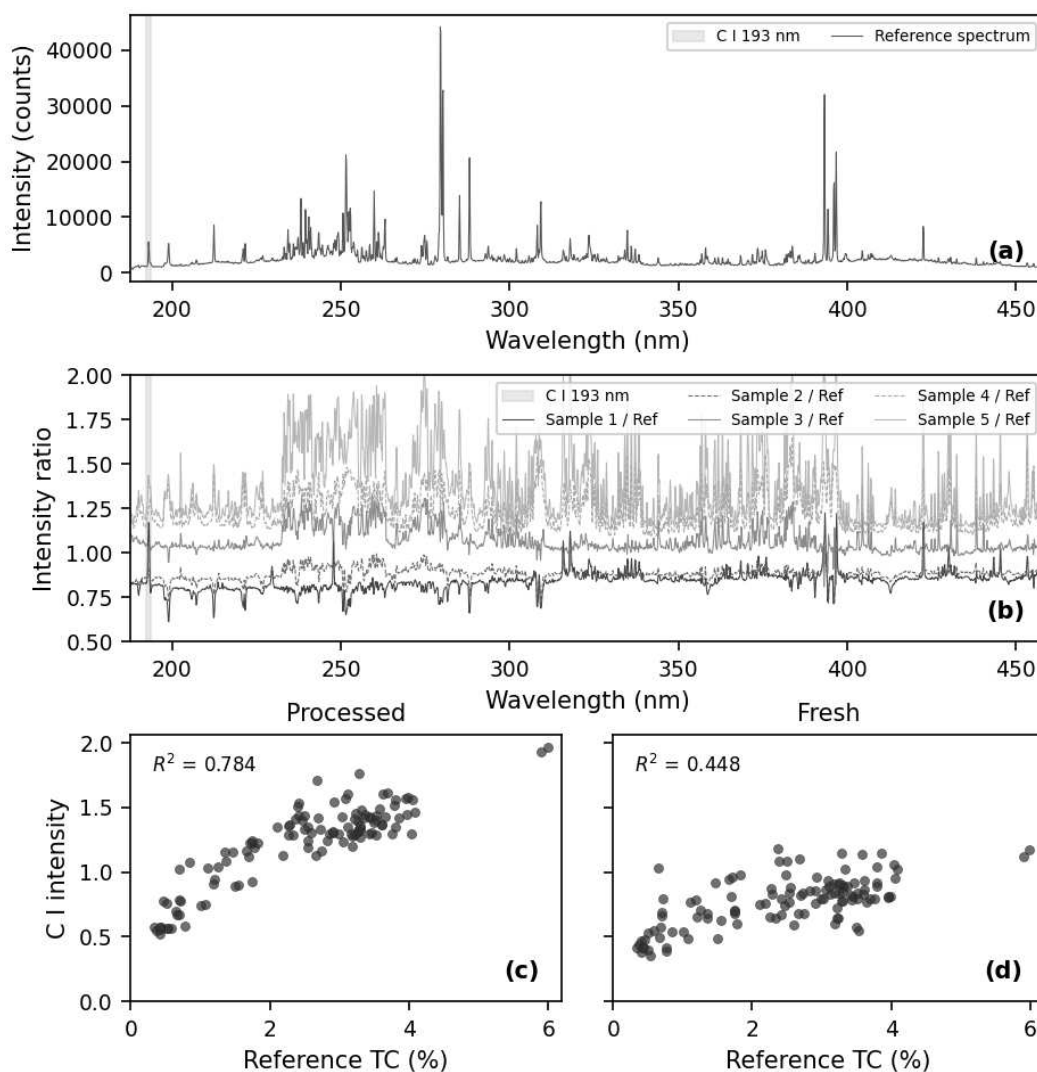


Figure 1. An example spectrum (a) compared to 5 other spectra with the same approximate carbon concentration (3.28 % - 3.43 %) (b) and the intensity of 193.1 nm C I line as a function of reference carbon concentration for all constant-humidity, milled samples (c) and varying humidity, non-milled (d) samples.

- A – Ball-milled samples at near constant humidity (N = 114)
- B – Fresh (non-milled) samples with natural varying humidity (N = 114)
- C – Ball-milled samples with lab-controlled varying humidity (N = 341)
- D – Non-milled samples with lab-controlled varying humidity (N = 341)
- E – All humidity states pooled (N = 689)

Groups A and B were modeled with 5 different splits per model to evaluate performance more robustly. Thus, the performance metrics presented for these groups are calculated by pooling all the prediction results from different splits together and then calculating the metrics for the model.

As can be seen from Supplementary Tables S1 and S2, Kernel Ridge was found to have the best carbon and humidity prediction power in our data set, followed by SVR and PLS. A scatterplot of the moisture content KR prediction for sample group E is shown in Fig. 2. We reached an excellent agreement with gravimetric moisture analysis with a coefficient of determination (R^2) value of 0.95 and a Ratio of Performance to Inter-Quartile distance (RPIQ)³¹ value of 7.6. Considering total carbon concentration, using the predicted moisture as a feature seems to improve model performance in most of the varying humidity cases (B-E): Of 24 models, if humidity is included, the value R^2 increases in 15 cases, decreases in 5 cases and stays

the same in 4 cases. Similar observation can also be made for other accuracy metrics. For the KR models, the difference was statistically significant only in sample group B ($n = 115$). Its Mean Absolute Error (MAE) values were 0.36 % TC vs 0.30 % TC for direct TC calibration and moisture-to-carbon predictions, respectively. Using the paired t-test on the absolute error values, the mean Δ of error was 0.06 % TC with 95 % CI from 0.018 to 0.098 % TC and a p -value of 0.005 when comparing the approaches.

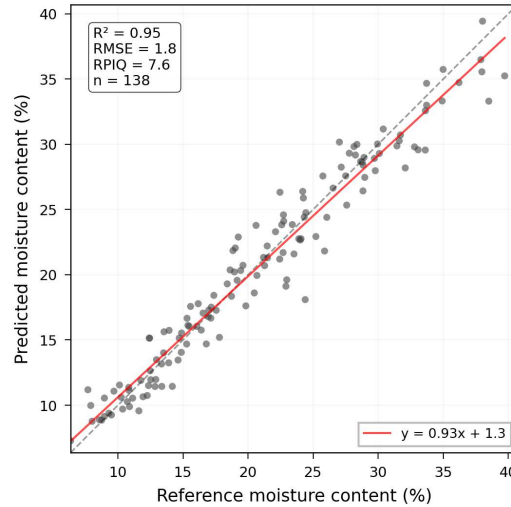


Figure 2. Kernel Ridge regression model test results for all samples with varying moistures (w_{H_2O}/w_{dry}).

The carbon concentration prediction results achieved with the KR model are shown in Table 1 for all moisture groups. In these results, the predicted moisture content was used as a feature in models B-E. In general, KR models from all sample groups show excellent prediction power. The best results are achieved with the constant-humidity sample group (A), with an R^2 value of 0.95, MAE 0.19 %-units and Root Mean Squared Error (RMSE) 0.27 %-units for the pooled test results ($n = 5 \times 23$). The results of this case are presented in Figure 3. The second best results are achieved when all varying-humidity samples are grouped together (group E), which leads to an R^2 value of 0.92, MAE of 0.26 %-units and RMSE of 0.34 %-units for the test set ($n = 138$). Thus, the results are best when the humidity is kept constant or the amount of data is increased. These results are expected: Humidity is shown to affect the emission intensity (Fig. 1), thus keeping it constant yields better results in prediction. Furthermore, the effect of varying humidity can be compensated for by increasing training data, which is shown as an increase in overall prediction power when all samples are included.

Table 1. The results of Kernel Ridge regression per calibration scenario. The results of group A have been conducted without moisture content as a feature, and in the other groups the moisture content has been predicted first and then used as a feature. The table includes the Coefficient of determination (R^2), Root Mean Square Error (RMSE), Mean Absolute Error (MAE), Mean Signed Error ($\bar{r}$) and the Ratio of Performance to Interquartile Distance (RPIQ). Positive ($\bar{r}$) indicates overprediction.

Calibration scenario	R^2	RMSE	MAE	$\bar{r}$	RPIQ
A – ball-milled, ≈ 15 % MC ($n = 5 \times 23$)	0.95	0.27	0.19	−0.016	7.1
B – fresh, natural humidity ($n = 5 \times 23$)	0.91	0.39	0.30	+0.006	5.0
C – ball-milled, varying humidity ($n = 69$)	0.86	0.44	0.31	+0.029	4.3
D – non-milled, varying humidity ($n = 69$)	0.86	0.43	0.31	+0.022	4.3
E – all states pooled ($n = 138$)	0.92	0.34	0.26	−0.084	5.9

The performance of sample groups B-D brings interesting observations: First, when comparing groups C and D, sample processing seems to have no significant effect on model performance under the lab-controlled humidity (mean Δ of error 0.00 % TC, 95 % CI from −0.07 to +0.07 % TC). Interestingly, measurements with the least sample processing and a limited amount of data (B) slightly outperform varying moisture sets (C and D), which have three times more training data; however, this difference is not statistically significant. When considering field-deployability, the performance of group B is key, as it is closest to soils measured on site. The results of groups B and E are shown in Figures 4 and 5, respectively.

Mean absolute errors by USDA texture–site group³² are shown in Table 2, which also lists the sampling site code for each texture class. The sites are described in more detail elsewhere³³. Because the samples are split into small per-cell groups,

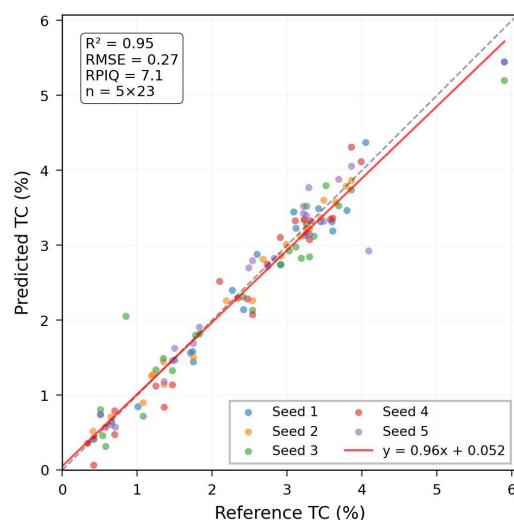


Figure 3. Kernel Ridge regression model for samples with constant humidity. 5 different splits are illustrated with different colors to ensure seed-independent robustness of the model. Metrics were calculated by pooling the test results together for all seeds.

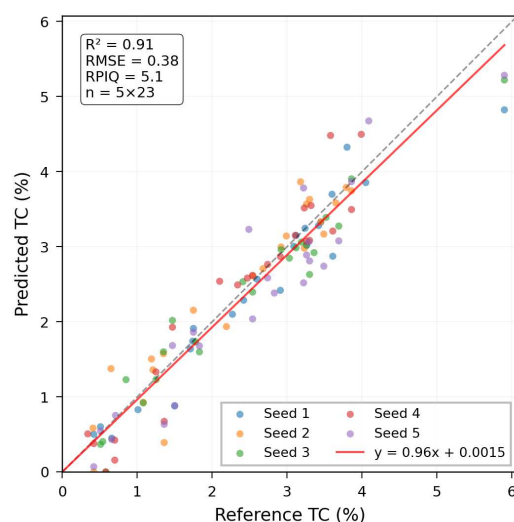


Figure 4. Kernel Ridge regression model for fresh samples with natural varying humidity. 5 different splits are illustrated with different colors to ensure seed-independent robustness of the model. Metrics were calculated by pooling the test results together for all seeds.

these values were not subjected to statistical testing and should be interpreted as observational trends only, not as statistically significant differences. In the constant-humidity group, clay (C) shows the largest error. For clay, the natural-humidity group gives equal results compared to the processed one, the 0.02 %-unit difference between the groups being plausibly within noise. Overall, the error appears more site-specific than texture-specific. The largest mean error is observed at the SE site (SiL / SiCL), whose texture class is close to that of the PU (SiCL) site, which has the second-lowest error. The SE site also contains outlying TC points at 5.90 and 5.99 % TC. As the next-highest values fall below 4.10 % TC, prediction of these two points suffers from sparse training data, which elevates the error. At the sites with the most observations (MÄ, JO, PA, LI, RU, PU) the mean error is close to that of the whole dataset, whereas the sites with fewer observations (SE, PI) deviate further. This pattern suggests that random error is a dominant contributor across the dataset, and that no firm conclusions can be drawn about any individual soil type being more error-prone from these results.

The errors separated on different depth horizons are presented in Supplementary Table S3. Smaller variation of error

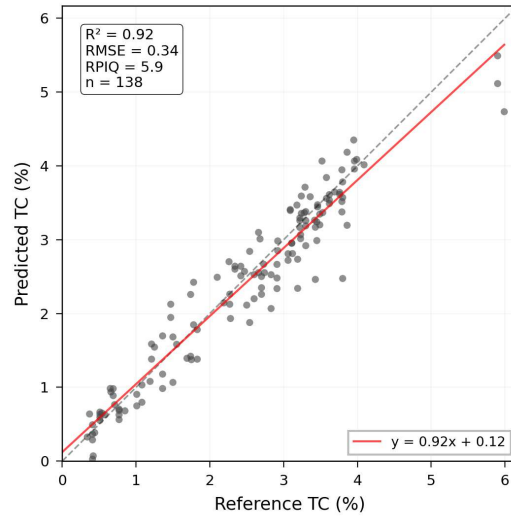


Figure 5. Kernel Ridge regression model for all samples with varying humidity.

was observed with varying depth than with the texture or site. Not a single horizon dominated the errors for each calibration scenarios, and the average error of each depth varied only between 0.24 % TC to 0.29 % TC.

Table 2. Mean absolute Kernel ridge residual (% TC) by calibration scenario (rows) and USDA texture–site (columns: bold texture over site abbrev). Scenario A uses a TC-only model; B – E use moisture-as-a-feature models. Cell entries are MAE (% TC) with n in parentheses. Bold marks the largest residual within each scenario row.

Calibration scenario	C MÄ	SiL / SiCL SE	SCL / C JO	C / SiC PA, LI	SiL RU	SiCL PU	C / CL PI
A – ball-milled constant ≈ 15 % moisture	0.32 (20)	0.29 (12)	0.13 (19)	0.13 (18)	0.17 (21)	0.15 (20)	0.22 (5)
B – fresh (non-milled) natural humidity	0.30 (20)	0.59 (12)	0.25 (19)	0.30 (18)	0.26 (21)	0.20 (20)	0.31 (5)
C – ball-milled lab-controlled varying humidity	0.39 (9)	0.35 (4)	0.27 (10)	0.29 (16)	0.40 (12)	0.31 (10)	0.17 (8)
D – non-milled lab-controlled varying humidity	0.35 (9)	0.46 (4)	0.39 (10)	0.36 (16)	0.29 (12)	0.20 (10)	0.17 (8)
E – all humidity states pooled	0.27 (18)	0.38 (16)	0.34 (26)	0.26 (21)	0.26 (16)	0.18 (24)	0.13 (17)
Weighted mean (n -weighted across A–E)	0.31 (76)	0.41 (48)	0.27 (84)	0.27 (89)	0.26 (82)	0.20 (84)	0.17 (43)

Discussion

The study aimed to evaluate whether LIBS, supported by vis–NIR diode reflectance and machine-learning models, can reproduce reference soil carbon concentration and moisture content with sufficient accuracy to serve as a faster, less labour-intensive alternative to the conventional methods. Dry combustion and gravimetric analysis were used as the reference methods for carbon concentration and moisture content, respectively, against which the LIBS-based predictions were benchmarked. Carbon concentration is the first measurement required for carbon stock determination, which additionally requires knowledge of the soil bulk density.

Six models were evaluated to predict the total carbon (TC) and moisture content (MC) of soils with varying sample processing and humidity conditions. The evaluated models were Kernel Ridge (KR), Support Vector Regression (SVR), Partial Least Square regression (PLS), Random Forest (RF), Neural Network (NN) and XGBoost (XGB). For our selection of samples and features, KR consistently outperformed the other models, closely followed by SVR and PLS. The best results were achieved with ball-milled constant humidity samples with an R^2 value of 0.95 and an RPIQ of 7.1 for KR. Good results were also achieved when measuring samples with natural humidity and a small amount of sample preparation ($R^2 = 0.91$ and RPIQ = 5.0).

This sample set is the most important when considering field-deployability, and thus the results show good field capability of the measurement system. Compared to earlier studies^{17,34–36}, the performance metrics presented here are above normal, despite the fact that most of the results are achieved with varying moisture. Previous research³⁵ labels RPIQ values above 3.7 as excellent, and this threshold is exceeded with all of the calibration approaches for KR models. The authors note that the high RPIQ values are affected by the wide spread in TC-% distribution, which, when connected with a low RMSE, drives the values higher³¹.

One aim of the study was to compare the prediction power of ball-milled samples with samples with less processing. We found that for our measurement system, ball milling does not play a role in prediction power, at least in the lab-controlled varying humidity sample sets. The results for the best performing model for these sample sets are almost identical (0.86 vs 0.86, 0.31 vs 0.31 and 0.44 vs 0.43 for R^2 , MAE and RMSE, respectively). Traditionally in LIBS, sample processing requires drying, sieving, and pressing a pellet from the sample with high pressure^{17,34–36}. Based on our data we propose that these steps can be mainly omitted, which would significantly streamline the analysis, saving on the total costs, and enabling on-site measurements. However, measurements with constant humidity still yielded the best results with a statistically significant difference between the errors of sample sets A and B (mean Δ of error 0.10 % TC with 95 % CI from 0.05 to 0.15 % TC and a p-value < 0.001).

Compared to the prediction of the carbon concentration, the prediction of moisture content needs more data to reach excellent metrics: When using only fresh samples with $N = 114$ (sample group B), the prediction power of the moisture content for KR model is moderate ($R^2 = 0.70$, MAE = 2.1 % MC, RMSE = 3.1 % MC). With the complete data set (sample group E), excellent performance is reached with an R^2 value of 0.95, MAE 1.4 % MC, RMSE 1.8 % MC and an RPIQ of 7.6. Because the results of LIBS measurements aimed at predicting the moisture content are unavailable in the literature, we compared these results to more traditional reflectance spectroscopy. The comparison shows that also the moisture content prediction performance falls in the above-average range^{37,38}.

Another goal was to compare whether predicting the moisture content first and then using that as a feature explicitly in the carbon prediction model brings any gains to the prediction power. The gain is visible in the sample sets with varying moisture, with statistical significance in sample set B. However, as one could expect, the constant humidity case (A) has no benefit from it. It was still tested as well, since the constant humidity sample set also had some variation in humidity, even though the aim of the set was to achieve as constant humidity as possible. It is possible that the C-D sample sets have limited gains from the humidity prediction because they already include more training data with the same soil samples including different humidity conditions. In sample set B, the gains from the humidity prediction as a feature are 0.86 to 0.91, 0.36 to 0.30 and 0.47 to 0.39 for R^2 , MAE (%) and RMSE (%), respectively.

Soil-class-specific error analysis indicates that the prediction error is less governed by texture class than the sampling site. The largest error was found to be in the site that has the widest range of TC %, especially considering the higher carbon values: the SE site includes values of 5.90 and 5.99 % TC while the next highest values are below 4.1 % TC. As machine learning methods rely on training data, careful attention has to be paid on building the training set to avoid the tendency of extreme values predicted towards the mean of the whole dataset. In other words, it is necessary to include the edges of the target variable distribution in the training set as well as possible. In our case where agricultural soils were analyzed, it is noteworthy that they represent the finest soil particle size classes, and thus, for further generalization it is necessary to analyze a wider range of soil types spanning from clay soils to sandy soils.

We have demonstrated the feasibility of Machine Learning methods for Vis-NIR-reflectance assisted LIBS measurements to predict soil carbon concentrations and moisture content. From a sample set of 116 individual soil samples, we were able to predict the carbon concentrations with good accuracy with splits with fewer than 100 samples in the training set. Considering that the samples covered six USDA texture classes and 8 spatially varying sampling sites, the amount of reference material needed vs. prediction accuracy is favorable compared to the more traditional reflectance spectroscopy methods^{9–11}, which require large regional libraries. A promising result is that carbon could be efficiently predicted from sample set B even with minimal sample processing and with varying, natural moisture. Furthermore, good results were achieved for the moisture prediction. To achieve excellent results, moisture prediction requires more reference data than carbon prediction.

The dry-combustion reference used here quantifies total carbon rather than soil organic carbon (SOC). In the studied acidic boreal mineral cropland soils the inorganic carbon fraction is expected to be negligible, so total carbon closely approximates SOC^{33,39}. Where this assumption does not hold, LIBS has been shown capable of discriminating organic from inorganic carbon^{40,41}, providing a route to direct SOC determination. Soil samples often contain materials such as rocks, coarse litter and plant roots that are not considered part of the soil organic carbon (SOC) pool and therefore need to be excluded from the analysis. Traditionally, these materials are removed by sieving or manual separation. Furthermore, converting SOC concentration measurements into SOC stocks requires knowledge of both the volume of the analyzed sample and its dry mass. To achieve the required precision and become a viable alternative to classical soil analysis methods, novel approaches must address all of these aspects simultaneously. The demonstrated moisture measurement eliminates the need for sample drying and thereby also enables bulk density determination under field conditions. Consequently, the next steps in method development

are to evaluate field sampling protocols and bulk density estimation against conventional soil coring and SOC determination methods with equivalent soil mass (ESM) calculations⁴² to achieve true on-site SOC stock determination.

Materials and methods

This chapter describes the soil samples, measurement hardware and data processing methods used in the study. Soil samples were collected from 8 different locations from Finnish agricultural lands, covering 6 USDA texture classes, two WRB Reference soil groups and carbon percentages between 0.34 to 5.99 % TC. The hardware was based on laser-induced breakdown spectroscopy (LIBS) assisted with LED-based vis-NIR reflectance. Feature extraction was conducted from the LIBS emission spectra by focusing on certain known elementary emission lines.

Soil samples and their preparation

A detailed table of the soil samples is presented in Table 3. The dataset included 116 samples from 8 different Finnish agricultural sites across Southern Finland, sampled in the years of 2019 and 2023. The samples were distributed across 16 plots and 5 depth intervals (0–10, 0–20, 10–20, 20–30 and 30–40 cm). They represented two WRB soil groups⁴³: Vertic Stagnosols (N = 70), Haplic Podzols (N = 28), and a transitional group of these two (N = 18). Furthermore, the samples covered 6 USDA texture classes: silt loam (SiL), silty-clay loam (SiCL), sandy-clay loam (SCL), clay loam (CL), silty clay (SiC) and clay (C). The total carbon in the samples varied from 0.34 to 5.99 m-%, spanning from the carbon-rich topsoils into the lower-carbon subsoils.

Table 3. Calibration sample set by sampling site. *n* counts unique physical samples per site (KOE/VER plot pairs × five depth intervals, 0–10 to 30–40 cm). USDA gives the site texture class (SiL = silt loam, SiCL = silty-clay loam, SCL = sandy-clay loam, CL = clay loam, SiC = silty clay, C = clay). TC and MC_{fresh} columns give empirical min–max ranges of total carbon (% w/w) and gravimetric moisture content (% w_{H_2O}/w_{dry}) for field-moist material.

Site	<i>n</i>	WRB Reference Soil Group	USDA texture	TC (% w/w)	MC _{fresh} (%)
JO	18	Haplic Podzols / Vertic Stagnosols	SCL / C	0.41 – 3.49	23.0 – 44.8
LI	10	Vertic Stagnosols	C / SiC	0.34 – 2.70	28.1 – 36.9
MÄ	20	Vertic Stagnosols	C	0.77 – 4.09	21.3 – 37.4
PA	12	Vertic Stagnosols	C / SiC	0.37 – 3.45	25.1 – 38.5
PI	10	Vertic Stagnosols	C / CL	0.51 – 3.96	29.2 – 34.9
PU	18	Vertic Stagnosols	SiCL	0.51 – 3.99	21.7 – 41.1
RU	18	Haplic Podzols	SiL	0.42 – 2.54	18.1 – 32.4
SE	10	Haplic Podzols	SiL / SiCL	0.41 – 5.99	24.1 – 40.0
Total	116	—	—	0.34 – 5.99	18.1 – 44.8

The 116 samples were divided into 2 subgroups: ball-milled and fresh samples. All of the samples were first homogenized by hand using a spoon and knife and the roots and stones removed by hand. Then, the samples were separated by placing a part of the sample into a sealed plastic bag and into the freezer at -20 °C. The other part was dried in an oven at 30–40 °C for approximately 7 days, after which it was ground in a ball mill into a fine powder using stainless steel containers. A part of the ground sample was then used to measure the reference values using a LECO 828 analyzer (LECO Corporate, MI, US). Due to too little soil material (< 5g), one sample was omitted from each sample set (milled and non-milled). The counterparts of the omitted samples were omitted from the other set to keep the measurement sets comparable in the analyses.

Of both sample sets, we prepared and measured 5 different combinations of humidities to demonstrate different calibration scenarios of the vis-NIR assisted LIBS analysis. The aim was to produce different datasets that ranged from a constant-humidity laboratory case to mimicking on-site measurements with as little sample preparation as possible. Another motivation was to investigate whether ball-milling has a significant effect on carbon prediction power compared to the less processed sample set. Thus, the prepared humidity sets were:

- A - Ball-milled samples with a constant moisture content of 15 % m_{H_2O}/m_{dry} ;
- B - Fresh samples measured directly from the freezer with their natural, varying humidity;
- C - Ball-milled samples with a lab-controlled varying humidity;
- D - Non-milled samples with a lab-controlled varying humidity;
- E - All samples with varying humidity bundled together.

In groups C and D the samples were dried, humidified into a known moisture content (MC) and measured three times per sample, leading to three different moisture states per sample. To ensure humidity variance for each sample, the humidities were stratified in such a way that for the three rounds the MC targets were 10 - 17%, 17 - 27% and 27 - 37%. An exception was made in the non-milled sample group, as there the natural humidity was used as one humidity group, as its humidities were mostly within the 27 - 37 % MC range.

LIBS measurements supported by vis-NIR diode reflectance

The measurements were conducted utilizing a combination of Laser-induced breakdown spectroscopy (LIBS) and LED-assisted vis-NIR reflectance measurements with an in-house instrument (Cense Analytics Oy, Finland). The fundamentals of LIBS and vis-NIR reflectance are presented in the literature elsewhere^{44,45}. Before measurement, the surface of each sample was smoothed using a manual toggle press. The samples were measured as flat 30 mm diameter pellets with a mass of ca. 5-10 g.

In the vis-NIR measurement, 5 different wavelengths of LED's were utilized to obtain information about the organic matter (OM) and moisture content of the sample. The wavelengths used were matched with known absorption features of OM and water in the vis-NIR wavelength range (450-2000 nm). Each wavelength was used in turns, and a one-pixel detector was used with a focusing lens to measure the reflectance values of the soil surface. Each sample was measured from six different positions to ensure a representative view of the sample surface. A schematic figure of the arrangement is presented in Figure 6.

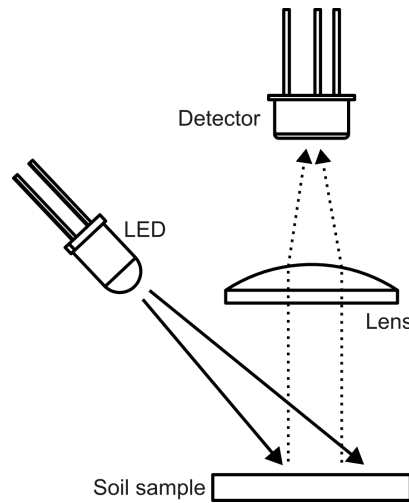


Figure 6. A schematic figure of the LED-assisted reflectance measurement arrangement. LEDs with different wavelengths were used to illuminate the sample in turns to resolve the reflectance of the sample surface with a few key wavelengths.

The LIBS measurement was conducted using a Q-switched Nd:YAG laser operating with a pulse energy of c.a. 40 mJ, wavelength of 1064 nm and a pulse frequency of 20 Hz. The pulses were focused onto the sample surface with a concave lens with 75 mm focal distance. The emission spectrum was collected with an optical fiber and directed into 2 spectrometers covering the ranges of 190 – 450 nm and 450 – 950 nm. The sample surface was scanned with the laser pulses by moving the sample in a plane along the focus spot, leading to approx. 350 laser shots/sample. A schematic figure of the LIBS measurement is presented in Figure 7.

Feature selection for quantitative analysis

The features were selected from both of the measurements presented in the earlier section. From the vis-NIR measurement, the reflectance-% of soil at each LED wavelength was used as a feature as-is. In addition, composite features for water absorption were calculated using a common background-normalized difference formula presented in earlier literature⁴⁶, in which for the feature value F :

$$F = \frac{R_{\lambda_{ref}} - R_{\lambda_f}}{R_{\lambda_{ref}}}. \quad (1)$$

In eq. (1), F is the calculated feature, $R_{\lambda_{ref}}$ is the reflectance of the background close to the absorption wavelength and R_{λ_f} is the reflectance at the absorption wavelength. Features like this were calculated for water absorption at 1450 nm and 1900 nm. Thus, total of 7 features from the LED measurement was used in the analyses.

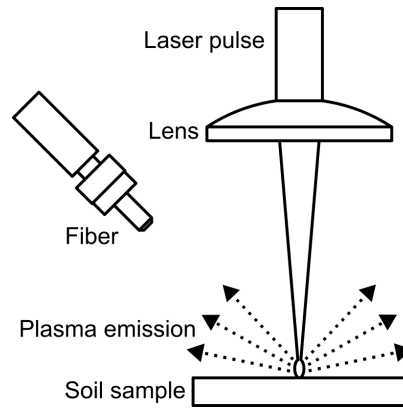


Figure 7. A schematic figure of the LIBS measurement arrangement. A pulsed laser with a wavelength of 1064 nm and an energy of 40 mJ, focused with a lens of $f = 75$ mm, was used to ablate the surface of the soil sample. The emission was captured with a fiber and directed into 2 USB-spectrometers.

Features from LIBS spectra were extracted by choosing a set of known elementary emission wavelengths. First, all of the 350 spectra from a given sample were filtered and averaged. The filtering was conducted based on the signal intensity at 193.1 nm, which is a known carbon emission line¹⁹. From the 350 spectra, the top 5 % of carbon intensities and the bottom 10 % intensities were omitted. The top filter ensures that any biological material is omitted from the spectra and the bottom filter omits any bad spectra due to cracks or deformations in the sample. The exact percentages were evaluated on empirical data to ensure that the filtering is adequate but not excessive. The remaining spectra were averaged. A baseline was fitted to each averaged spectrum using an asymmetrically reweighted penalized least squares (ARPLS⁴⁷) method, with a smoothing parameter of $\lambda = 10^4$. The spectra were then normalized by subtracting the baseline and then dividing with the baseline:

$$I_n = \frac{(I_{raw} - B)}{B}. \quad (2)$$

In eq. (2), I_n is the normalized intensity, I_{raw} is the raw emission intensity and B is the baseline. The normalization with the baseline enables extra robustness to possibly varying absolute signal levels that might occur due to varying measurement conditions, laser power fluctuation, optics contamination etc. Figure 8 illustrates the baseline correction and the chosen wavelengths for feature extraction.

Figure 9 illustrates the feature extraction flow from the chosen elements. As the main goal was to measure carbon concentration and moisture content, carbon emission lines at 193.1 nm, 229.7 nm and 247.8 nm and hydrogen emission line at 656.3 were processed in a more precise manner by Pseudo-Voigt peak fitting, while the other element lines were processed by a more simple approach. From the peak fits of carbon and hydrogen, the area, amplitude and peak width were used as features (Fig. 9 (a)). The other approach used for other elements was to simply choose a window of interest for each element and calculate the mean of the 5 highest values in the window (Fig. 9 (b)). This approach allows to mitigate the possible fluctuation between spectrometer pixel positions with respect to the emission maximum, and thus might increase the comparability between different peaks of the same element or between different nominally identical spectrometers.

A set of features were also extracted from the ARPLS baseline for each spectrometer, namely the mean, integrated area, number of peaks, variance, skewness, and kurtosis. In total, the elementary features consisted of 110 features, leading to a total of 122 LIBS-derived features when adding the baseline. The same set of features were used in the moisture content and carbon concentration models. In addition, carbon content models were also evaluated by integrating the predicted moisture content as a single feature into the model.

Moisture content as a feature

As shown in Supplementary Table S2, increasing the number of samples in the prediction of moisture content substantially increases the prediction power. To evaluate the benefit of predicted moisture inserted into the carbon analysis, one has two options: (i) use the same training set as is used for carbon prediction in the moisture prediction as well, or (ii) have a global, different model for the moisture. Both approaches come with their caveats: Approach (i) may lead to underestimation of the actual MC prediction accuracy, as it was noticed that moisture content predictions need more training data than carbon predictions. Approach (ii) – using a global model – may suffer from overfitting if the same samples are used to simulate the moisture content prediction power. As moisture content training data is inexpensive to achieve, we chose to use the approach

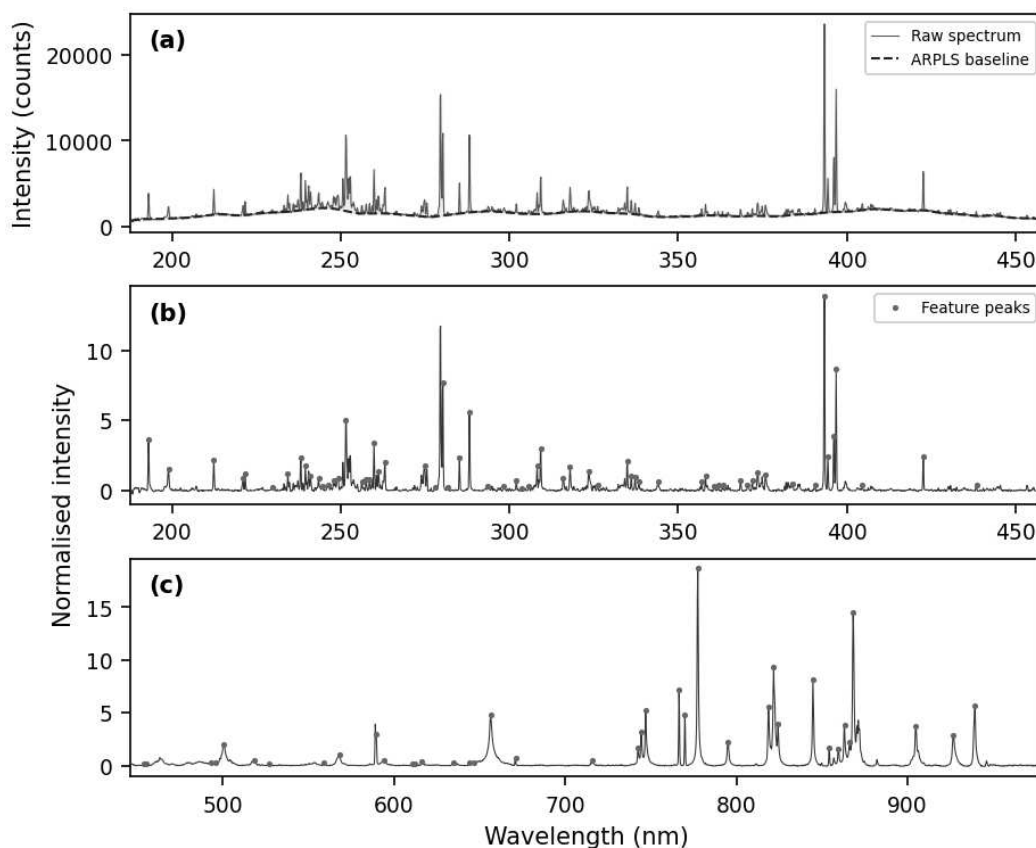


Figure 8. Illustration of the emission signal processing. The spectra were normalized with an ARPLS baseline and intensities of key elements were extracted from the normalized spectra.

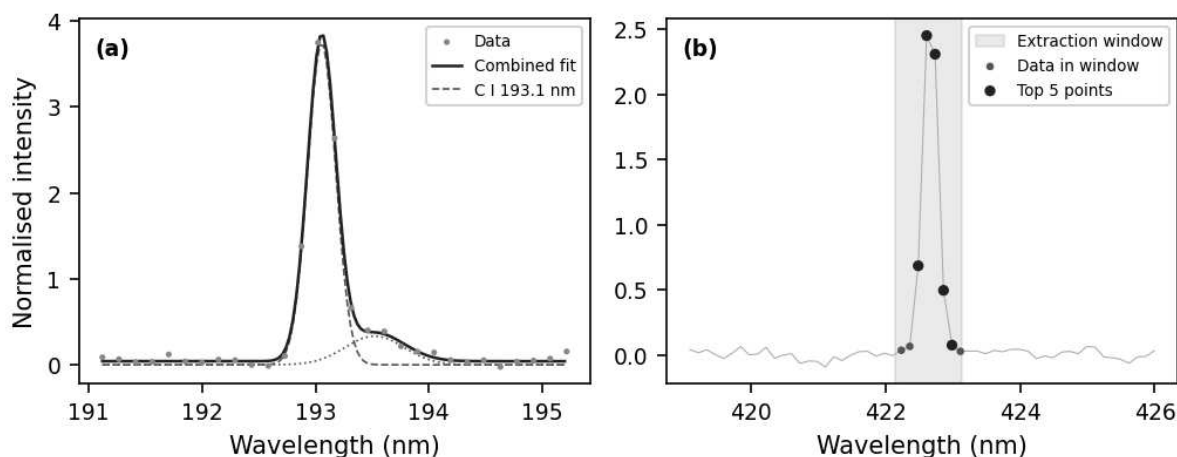


Figure 9. An illustration of the feature extraction from the chosen elements. Carbon and hydrogen were processed with pseudo-Voigt peak fitting (a), and other elements with a simple pixel intensity counting method (b).

(ii), but paid special attention to minimize overfitting. Thus, we used sample group E to train a linear SVR model and included all samples into its training set. A linear SVR was chosen because it was found to be least prone to overfitting with our set of data. Performance metrics for the linear SVR model when predicting the entire sample population – the same on which it was trained – were 0.94, 1.5 %-units and 2.0 %-units for R^2 , MAE and RMSE, respectively. Thus, the metrics were slightly lower than the held-out test set of the best KR model, which indicates a non-optimistic MC prediction performance. The SVR model

was then used in all the moisture predictions that were inserted as features into the TC prediction models.

Model construction and evaluation

As shown in Fig. 1, univariate calibration from a single LIBS emission line yields poor prediction accuracy, especially when humidity varies. In addition to moisture, matrix effects from mineralogy, texture, and elemental composition alter the intensities of the target carbon lines and, concurrently, the relative emission of other elements in the plasma. LIBS spectra are therefore high-dimensional and mutually correlated: changes in sample conditions shift many peak and baseline features together rather than isolating a single analyte signal. Machine-learning methods are well-established data-driven regression models that can combine these inputs and capture the nonlinear interactions among matrix components and moisture-related features. By learning how the emission features co-vary, multivariate models can represent couplings that are difficult to express in fixed linear univariate calibrations, which makes machine learning especially suitable for LIBS emission spectra combined with auxiliary reflectance data.

We selected six regression models: Partial Least Squares (PLS), Kernel Ridge Regression (KR), Support Vector Regression (SVR), Neural Network (NN), Extreme Gradient Boosting (XGB), and Random Forest (RF). These models were chosen to cover linear latent-variable methods, kernel-based nonlinear approaches, ensemble tree methods, and neural network architectures, enabling comprehensive comparison of their suitability for soil carbon concentration and moisture content prediction.

All input features were standardized to zero mean and unit variance prior to modeling. Log-transformation was applied to the target variable before optimization ($\log_{10}$ for total carbon, natural logarithm for moisture content). The dataset was divided into training (80 %) and test (20 %) sets using 10-bin quantile stratification based on the transformed target variable to ensure representative distribution across both sets. The splits were conducted deterministically in a way that the held-out partitions were identical for A and B and identical for C and D to ensure comparability between the different sample processing approaches.

Hyperparameters were selected using Bayesian optimization (Optuna⁴⁸) with the Tree-structured Parzen Estimator (TPE) sampler over 200 trials (50 for NN). The optimization objective was R^2 calculated on the original (back-transformed) target value scale. Each trial was evaluated using 5-fold cross-validation repeated 5 times (25 total folds), providing robust performance estimates while accounting for fold variability. The search ranges of the hyperparameters per model can be found in Supplementary Table S4 and the performance of the cross-validation from Supplementary Table S5.

To prevent data leakage, all preprocessing steps—including feature scaling and feature selection—were performed independently within each cross-validation fold. For feature selection, mutual information regression was computed separately for each fold's training data to rank features by relevance. The number of top-ranked features to retain (min. 5) was optimized jointly with other hyperparameters. This leakage-free approach ensures that validation performance accurately reflects model generalization capability.

The final model for each algorithm was trained on the entire training set using the best hyperparameters identified during optimization. Model performance was evaluated using the test set with three metrics on the original scale: coefficient of determination (R^2), root mean squared error (RMSE), and mean absolute error (MAE). The Ratio of Performance to Interquartile Distance (RPIQ)³¹ values were also calculated for the best models.

Statistical evaluation between different sample preparations and calibration approaches was performed with the two-sided paired t -test using the absolute errors of each paired sample. The t -tested calibration scenarios were:

- Scenario A vs B
- Scenario C vs D
- Same sample scenarios with and without predicted moisture as a feature

From the t -test, the p -value and the mean Δ of the error was calculated, including its 95 % confidence intervals.

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Author contributions statement

P.H. Drafted and wrote the majority of the article, generated the images, and participated in the data processing. Designed and supervised the measurements.

W.X. Participated in the writing and data processing.

J.H. Participated in the writing, analyzed reference samples, and provided information on the soil samples.

J.V. Participated in the writing, managed the funding acquisition, and supervised the work.

Data availability

The data generated during and/or analyzed during the current study are available from the corresponding author(s) on reasonable request.

Competing interests

The authors declare the following competing interests at the time of submission:

J.V. is a co-founder and a shareholder of the company Cense Analytics Oy.

P.H. is a shareholder and an employee of the company Cense Analytics Oy.

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