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Using loss-on-ignition to estimate total nitrogen content of mangrove soils

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ABSTRACT

Loss-on-ignition (LOI) has been widely used to estimate soil organic carbon (OC) content for coastal wetland soils, owing to recent interest in 'blue carbon' systems. Comparatively less attention has been paid to soil nutrient retention, specifically total nitrogen (TN), despite being a historically limited resource that influences C cycling and aquatic ecosystem health. A single conversion equation is available to estimate soil TN content from LOI, derived from salt marshes. This study investigated the utility of creating TN conversion equations from LOI data for application in mangrove soils to improve global understanding of their role in TN interception and sequestration. A dataset of 1275 mangrove soil samples from 17 regions in the western hemisphere was used to create one general equation, two equations pertaining to sedimentary settings, and equations for each of seven coastal environmental settings (CES) that differed in mineral sediment provision, geomorphology, and hydrology. Soil mean LOI and TN ranged from respective lows of 9.5% and 0.18% in terrigenous estuaries to respective highs of 51.4% and 1.25% in carbonate estuaries. The ability of LOI to predict TN in mangrove soils was strong, with an R^2 for the general equation of 0.88, and several regional equations exceeding 0.90. Total N represented between 1.1 and 3.7% of LOI. Differences in equations were minimal between mangrove soils and a previous equation for salt marsh soils, and between carbonate and terrigenous sedimentary settings. Significant differences were observed among CES. Deltas had the lowest TN:LOI (0.014), followed by terrigenous estuaries and open coasts (0.017 and 0.019, respectively). Terrigenous lagoons, carbonate estuaries, and carbonate open coasts had the highest TN:LOI (0.024 for all three). The study provides a decision tree to select an appropriate approach for estimating TN content of mangrove soils, offering an important tool for improving the understanding of the role of coastal wetlands in global TN sequestration.

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1. Introduction

The photosynthetic reduction of atmospheric CO_2 into organic carbon (OC), which drives the fundamental provision of energy for much of life on earth, requires at least 25 macro- and microelements (Kaspari and Powers, 2016). While nitrogen (N) is of particular importance due to its role in the synthesis of proteins and nucleic acids in living tissues (Novoa and Loomis, 1981), organic matter N represents only 0.002 % of all N on Earth (Vitousek et al., 1997). Nitrogen availability influences both primary and secondary production and is therefore an important control on C cycling and atmospheric greenhouse gas concentrations (including CO_2, CH_4, N_2O), and global warming. The coupling of N to primary and secondary production also influences coastal aquatic ecosystem health, and excess N availability contributes to algal bloom production, depletion of oxygen, fish kills, and potentially remineralization of previously sequestered C.

Soil total nitrogen (TN) includes both organic and inorganic forms (Fig. 1). Roughly 90 % or more of soil TN is organic, representing approximately 5 % of soil organic matter (SOM) mass, and is comprised of amino acids, amino sugars, as well as both acid-insoluble and hydrolysable nitrogen (Fig. 1, Schulten and Schnitzer, 1997). Forms of inorganic nitrogen (IN) include mineral-occluded N or the exchangeable and soluble forms of NH_4^+, NO_3^-, and NO_2^-. Mineral forms of N can be relatively transient and undergo rapid changes which are facilitated by translocation, diffusion, microbial mineralization and immobilization, and uptake by plants (Bremner. 1965; Stevenson 1982; Schulten and Schnitzer, 1997). However, IN may occasionally be geologically long-lived, such as NH_4^+ bound as a substitution for potassium in silicate minerals or in the form of NH_4^+ and NO_3^- salts (Holloway and Dahlgren 2002).

Over the past three decades, considerable attention has been given to the SOM stored in coastal blue carbon ecosystems (e.g., mangroves, salt marshes, seagrasses, tidal freshwater marshes, and forested wetlands) (Adame et al., 2024; Kauffman et al., 2020; Donato et al., 2011; Sanderman et al., 2018; IPCC, 2019; Rosentreter et al., 2023), as evidenced by the sharp increase in publications mentioning soil C (4,565 references from 1991 to 2022) due to its important relevance to global climate (Fig. 2). In comparison, 3,364 references mention soil N in the same time period (Fig. 2). There is little available information about the global-scale ability of mangroves to sequester N runoff from the land before it reaches coastal waters where it may otherwise contribute to eutrophication, coastal anoxia, fish kills, and algal blooms; only a handful of studies have directly measured N stocks or burial rates within mangrove soils. There is also uncertainty about the effect that sequestered N may have on biogeochemical cycling and stabilization of blue carbon in coastal soils as a result of linkages between N and C cycling (Voss et al., 2013; Pajares and Ramos, 2019; Herbert, 1999). This has catalyzed a growing body of research quantifying N stocks and fluxes in coastal wetlands (Reis et al., 2017a; b; Alongi 2020; Pérez et al., 2021; Wigand et al., 2021).

Loss-on-ignition is the one of the most commonly used methods for quantifying C worldwide (Hoogsteen et al., 2015). There is an abundance of LOI values present in the coastal wetlands literature, driven in part by the recommendation of soil OC accounting manuals (Howard et al., 2014) to use LOI as a low-cost alternative to elemental analysis (at virtually no cost for LOI analysis vs. \$10–20 per sample for elemental analysis) for estimating soil OC content. Although dozens of peer-reviewed equations exist to estimate OC from LOI (Pribyl 2010; Bennett and Chambers, 2023; Breithaupt et al., 2023), to our knowledge Craft et al., (1991) provide the only published equation that uses LOI to estimate TN in salt marsh soils: $TN = 0.020 \times LOI + 0.00005 \times LOI^2 - 0.018$. In the last 32 years, there have been 163 citations of the Craft et al., (1991) OC conversion equation, but only 3 citations for the TN conversion (Fig. 2B).

The increasing usage of LOI to estimate coastal wetland OC (i.e., blue C) in the past decade and a half represents an opportunity to broaden the global estimation of TN in these environments. Although the relationship between LOI and organic N would almost certainly be stronger, our focus here is on the relationship of LOI and TN because TN is almost always measured simultaneously with TC or OC on an elemental analyzer (EA), and elemental stoichiometry of wetland biomass and soils is stated in terms of OC:TN:TP. The primary objective of this research was to investigate the relationship between LOI and TN in mangrove soils to a) determine how robust the relationship is, and b) provide a low-cost alternative to elemental analysis for estimating soil N content of mangrove soils. Such an equation would be widely useful when applied to new and existing published data for increasing knowledge about coastal wetland soil N. The cost savings of using LOI instead of an

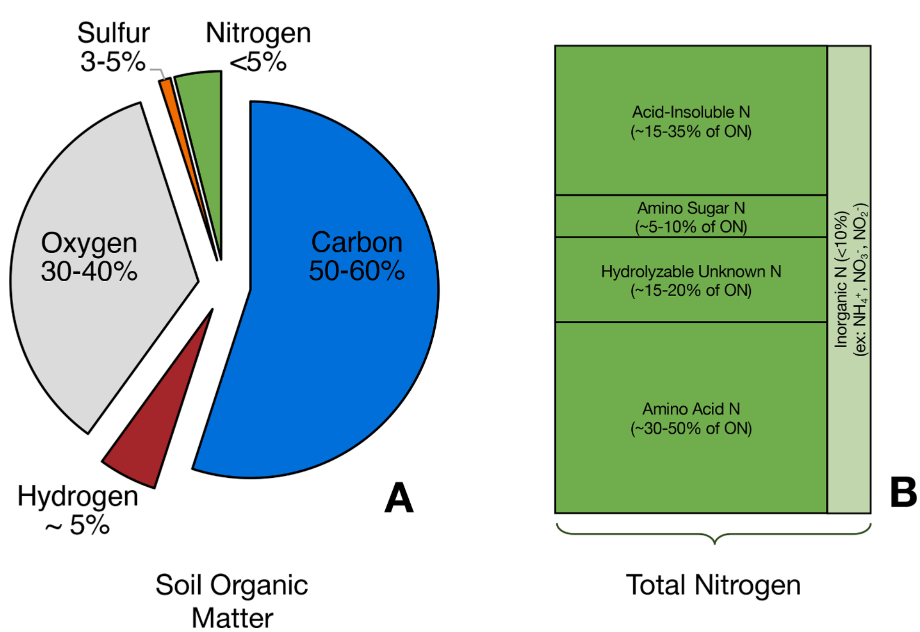

Fig. 1. A) Proportions of five most abundant elements (by mass) that comprise organic matter (SOM), operationally quantified by loss-on-ignition (LOI) in highly organic soils (Rutherford et al., 1992), and B) the relative proportions of organic and inorganic N constituents of Total N (adapted from Stevenson, 1982; Schulten and Schnitzer, 1997).

EA provide a means for increasing soil TN estimates from regions in many of the global developing economies where mangroves dominate the coastline. Additionally, although lab costs in developed countries may be less of a barrier to analysis, projects with large sample numbers, including those conducted by state or regional coastal monitoring entities without large analytical budgets, will benefit from a framework for estimating soil TN content. The secondary objective of this study was to evaluate whether the LOI-TN relationship in mangrove soils varies (1) from the relationship for salt marsh soils, and (2) according to coastal sedimentary and geomorphic settings characterized by differences in sediment composition and hydrology (Worthington et al., 2020; Breithaupt et al., 2023). For example, does the LOI-TN relationship differ among deltas, terrigenous open coasts, or carbonate estuaries? The framework establishing the usefulness of coastal environmental setting (CES) for differentiating processes and structure in mangrove forests (Thom, 1984; Woodroffe 1992; Woodroffe et al., 2016; Dürr et al., 2011; Balke and Friess, 2016; Twilley and Rivera-Monroy, 2009) has been used to reveal differences in forest structure and biomass (Rovai et al., 2021), soil OC stocks and OC:TN:TP stoichiometry (Rovai et al., 2018), OC burial rates (Breithaupt and Steinmuller, 2022), the LOI-OC relationship (Breithaupt et al., 2023), carbon dioxide and methane emissions (Rosentreter et al., 2023), and root production (Arnaud et al., 2023).

2. Methods

A multi-region dataset was compiled from 12 studies that reported LOI and TN data for mangrove soils between 26°S and 30°N in the western hemisphere (Fig. 3, Table S1). Each study collected multiple soil cores from mangrove wetlands with total core lengths ranging from 15 to 100 cm. Cores were sectioned into depth intervals ranging from 1 to 5 cm for a total of 1275 observations.

2.1. Loss-on-ignition and total N analysis

Each soil sample was dried until a constant weight was achieved, homogenized, and analyzed for percent organic matter using LOI (Table S1; Breithaupt et al., 2023). For the purposes of this work, LOI procedures were standardized across labs (550 °C for 3 h) to minimize operational differences in results. Total N was determined via elemental analysis using a Carlo Erba 1500 CN, an Elementar Vario Micro Cube CHNS Analyzer, or a PDZ Europa ANCA-GL (Table S1).

2.2. Classification of coastal environmental settings

Study regions were classified as one of seven CES following the framework developed by Worthington et al., (2020) and described in Breithaupt et al., (2023). A coastal environmental setting consists of a word-pairing, where the first term (terrigenous or carbonate) identifies whether a site's mineral sediment is primarily derived from terrigenous sources or is chemically precipitated autochthonously (Balke and Friess, 2016). The second term identifies the geomorphic setting (delta, estuary, lagoon, or open coast) and is derived from the naming conventions employed by Worthington et al., (2020) that primarily relied on the two-dimensional shape of a coastline for automated machine learning and mapping. As such, deltas are identified as a protrusion of land into the sea at river mouths, estuaries represent open, concave shapes where rivers meet the sea, lagoons represent enclosed bodies of coastal water, and open coasts represent non-enclosed and non-riverine coastlines. There are seven possible CES combinations, excluding "carbonate deltas" because a delta is formed by the accumulation of allochthonous material, something that by definition does not form in a carbonate depositional environment. While other coastal typologies exist, this CES typology is used here because it allows for standardized identification and comparison with a global map of mangroves, including the proportion of total global mangrove area represented by each CES. Most of our sites were classified using the typology and geographic information systems (GIS) layer of Worthington et al., (2020), however, a few sites were re-classified based on local knowledge. In situations where the site classification either a) did not exist because the region was not included in the global mangrove map, or b) differed from local knowledge, the typological assignment was made based on the regional expertise of co-authors (Table S2).

2.3. Statistical analysis

Differences in the fractions of organic sediment mass and TN content among sedimentary settings and CES were determined via a *t*-test (carbonate vs. terrigenous, $\alpha = 0.05$) and via a linear model (CES, $\alpha = 0.05$). This and all subsequent tests were computed in R (version 4.1.0; R Core Team, 2023) with RStudio (version 2023.03.1 + 446; Posit Team, 2023). Percent organic matter and percent inorganic matter datasets were checked for normality using the Shapiro-Wilk test.

Conversion equations were calculated using linear regressions taking the form $TN = m \times LOI + b$, where TN and LOI are both represented as a percent of dry soil mass. In addition to creating conversion equations for each of the 17 regions in our dataset, a general mangrove equation was

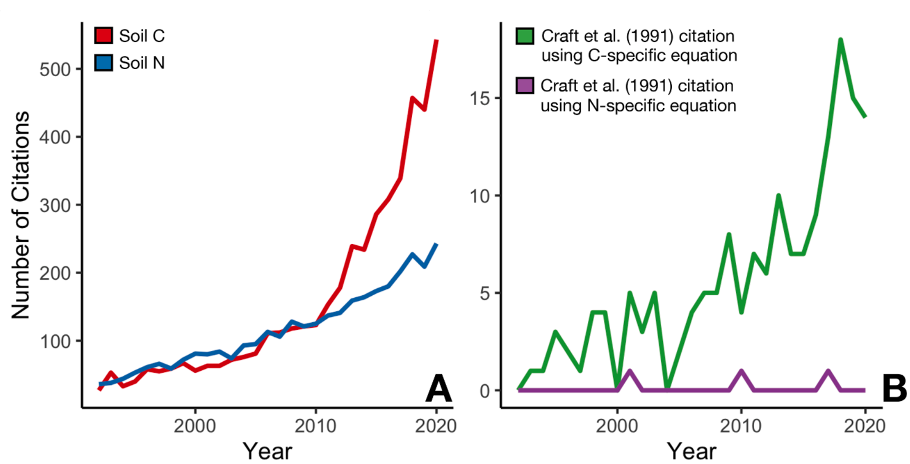

Fig. 2. Number of citations by year for A) publications about coastal vegetated ecosystems (i.e., mangroves, tidal marshes, or seagrasses) that mention soil C and soil N; and B) publications citing the Craft et al., (1991) conversion equations for estimating organic carbon or total nitrogen. Panel A results derived from a January 27, 2023 Web of Science search identifying publications from 1991 to 2022 based on the occurrence of topics "carbon" or "nitrogen" plus "mangrove", "marsh", or "seagrass". Data for panel B were similarly obtained from a Web of Science search but including a citation of Craft et al., (1991).

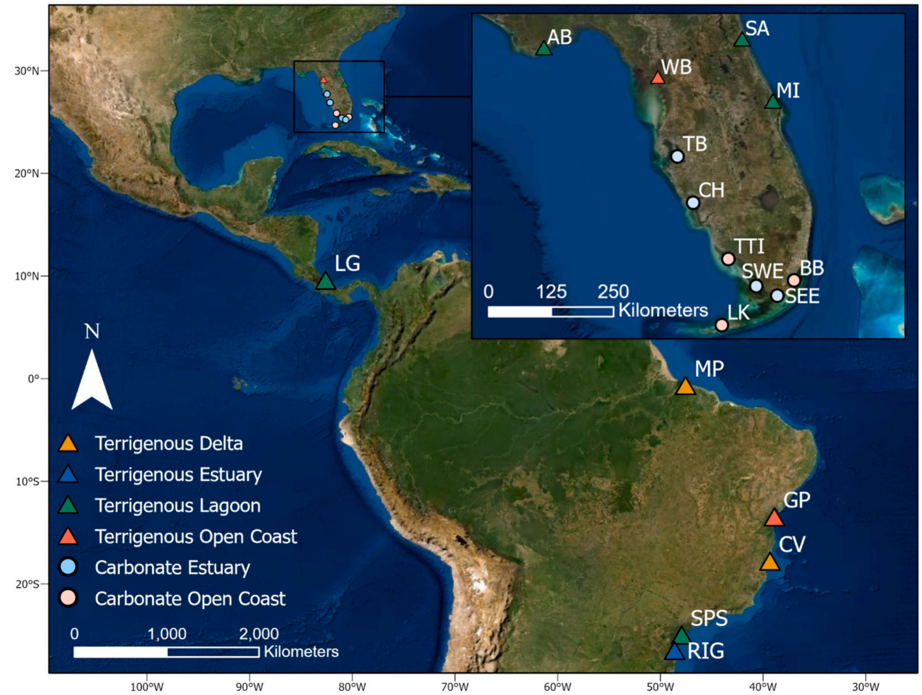

Fig. 3. A) Locations of regions used to compile the dataset. B) Inset of regions within coastal Florida. Colors on both panels denote the assigned coastal environmental setting (CES) for each sampling region.

produced using data from all regions, as well as equations for carbonate and terrigenous sedimentary settings, and the six combinations of sedimentary and geomorphic settings for which data were available in our dataset: carbonate estuaries and open coasts, and terrigenous deltas, estuaries, lagoons, and open coasts (Table 1). The ratio of TN to LOI (TN: LOI) was calculated for the dataset and was used for statistical comparisons. The TN:LOI data were log-transformed to meet the kurtosis and skewness assumptions of normality (Hair et al., 2010; Byrne, 2013). Heterogeneity of variance was determined using Levene's Test. One-way analysis of variance (ANOVA) was used to determine how log-transformed TN:LOI differed among CES. Post-hoc tests were performed using the package 'multcomp' (Hothorn et al., 2016) to determine whether significant differences existed among CES.

To test the performance of our three categorical equation types (general, sedimentary setting, and CES; Table 1), the 17 regional equations were used as standards for comparison. The R^2 and root mean square error (RMSE) values were calculated for the fit of each categorical equation type to the observed regional data. A decision tree for evaluating and recommending selection of an equation type was developed based on which equation type had the highest mean R^2 and lowest mean RMSE values for the 17 regions. All stated uncertainties represent 1 standard error unless otherwise stated.

3. Results

3.1. Mangrove soil TN and LOI content

Loss-on-ignition values of the dataset ranged from 1.04 to 87.13 % with a mean of 42.80 ± 0.68 % (Table 1). The mean LOI of carbonate setting soils was 50.68 ± 0.71 %, over two times greater than the mean LOI of 20.37 ± 0.87 % for terrigenous settings ($t = 27.039$, $df = 799.46$, $p < 0.0001$). Carbonate estuaries and open coasts had similar LOI values of 51.36 % and 48.58 % ($p > 0.05$). Loss-on-ignition percent differed among the CES (F value = 114.2, $df = 5$, $p < 0.0001$). Of the four terrigenous geomorphic settings, estuaries had the lowest mean LOI of 9.50 ± 0.94 %, and deltas, lagoons and open coasts ranged from 15.94 ± 0.92 to 24.09 ± 1.55 %.

Total N values of the dataset ranged from 0.03 to 2.83 % of dry soil mass with a mean of 1.01 ± 0.02 % (Table 1). Carbonate sedimentary setting sites had an average TN of 1.22 ± 0.02 % which was almost three times greater than the average of 0.43 ± 0.02 % for terrigenous setting soils ($t = 27.679$, $df = 757.84$, $p < 0.0001$). Total nitrogen percent was

Table 1
Details of the nine equations generated from these overall dataset (Eq. 1 is provided in the Introduction, from Craft et al., 1991). The ranges and means (±standard error) of loss on ignition (LOI) and total nitrogen (TN) values, as well as the slopes, y-intercepts (±standard error), and R^2 for the regression of TN vs. LOI for the entire dataset (general), carbonate and terrigenous sedimentary settings, and each coastal environmental setting (CES). n denotes sample size. For mean LOI, different superscripted lowercase letters indicate differences in carbonate settings vs. terrigenous settings, and among the six coastal environmental settings.

Equation	Data	n	LOI Range (%)	Mean LOI (%)	TN Range (%)	Mean TN (%)	Slope	y-intercept	R^2
1	General	1275	1.04–87.1	42.8 ± 0.680	0.030–2.83	1.01 ± 0.02	0.0237 ± 0.0002	−0.0029 ± 0.0001	0.88
2	Carbonate	943	1.78–87.1	50.7 ± 0.710 [a]	0.030–2.83	1.22 ± 0.02	0.0225 ± 0.0003	0.0766 ± 0.0002	0.83
3	Terrigenous	332	1.04 – 78.0	20.4 ± 0.870[b]	0.030–2.44	0.43 ± 0.02	0.0235 ± 0.0006	−0.0545 ± 0.0002	0.81
4	Carbonate Estuary (C_E)	712	1.78–87.1	51.4 ± 0.890 [a]	0.030–2.83	1.25 ± 0.02	0.0229 ± 0.0003	0.0720 ± 0.0002	0.86
5	Carbonate Open Coast (C_OpC)	231	12.3–77.5	48.6 ± 0.980 [a]	0.250–2.30	1.13 ± 0.03	0.0195 ± 0.0011	0.1864 ± 0.0006	0.58
6	Terrigenous Delta (T_D)	71	4.12–41.6	15.4 ± 0.920[b]	0.040 – 0.493	0.20 ± 0.01	0.0116 ± 0.0002	0.0199 ± 0.0001	0.74
7	Terrigenous Estuary (T_E)	36	1.04–23.4	9.50 ± 0.940[c]	0.030–0.380	0.18 ± 0.02	0.0172 ± 0.0018	0.0165 ± 0.0002	0.79
8	Terrigenous Lagoon (T_L)	160	1.13 – 78.0	24.1 ± 1.55 [bd]	0.040–2.44	0.58 ± 0.04	0.0232 ± 0.0008	0.0177 ± 0.0003	0.83
9	Terrigenous Open Coast (T_OpC)	64	7.57–44.7	22.2 ± 1.40 [d]	0.090–1.08	0.42 ± 0.04	0.0243 ± 0.0012	−0.1187 ± 0.0003	0.87

different among the CES (F value = 131, df = 5, $p < 0.0001$). Terrigenous deltas, estuaries, and open coasts had the lowest mean TN values, ranging from 0.18 – 0.42 %, followed by terrigenous lagoons (0.58 ± 0.04 %) and carbonate open coasts (1.13 ± 0.03 %); carbonate estuaries had the highest TN% of 1.25 ± 0.02 ($p < 0.0001$; Table 1).

3.2. Conversion equations

The slope of the general conversion equation derived from all samples indicated that TN represented 2.37 % of LOI mass ($R^2 = 0.88$; Table 1; Fig. 4A). Conversion equations for each of the 17 regions in this dataset (Fig. 5) indicated that TN as a percentage of LOI varied by over a factor of three, from 1.09 to 3.68 % (Fig. 5).

The slope of CES equations ranged from 0.0140 ± 0.0002 in terrigenous deltas to 0.0243 ± 0.0012 in terrigenous open coasts (Table 1). The y-intercepts for CES equations ranged from −0.0012 ± 0.0003 in terrigenous open coasts to 0.0019 ± 0.0006 in carbonate open coasts. The lowest R^2 for a conversion equation was 0.58, for carbonate open coasts (Table 1). All other conversion equations had $R^2 > 0.70$ (Table 1), and all presented equations displayed $p < 0.05$.

3.3. TN:LOI ratios

The TN:LOI was significantly different among CES ($p < 0.001$, F = 48.47) The mean TN:LOI for terrigenous deltas (0.014 ± 0.001) was lower than that of all other CES (Fig. 6). There was no difference between terrigenous open coasts and terrigenous estuaries, with mean TN:LOI values of 0.017 ± 0.001 and 0.019 ± 0.001, respectively (Fig. 6).

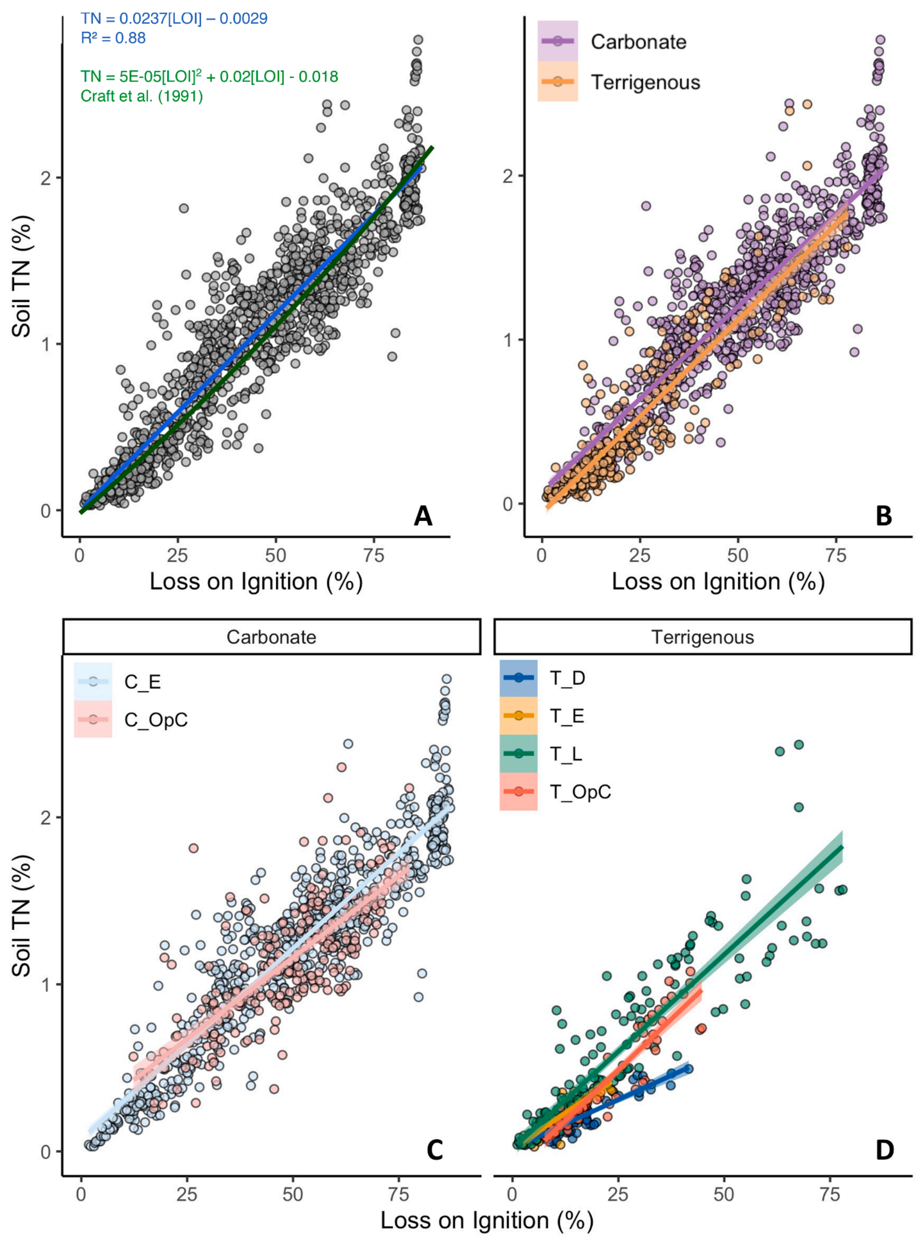

Fig. 4. Relationship between percent loss-on-ignition (LOI) and percent total nitrogen (TN) for A) all mangrove soil samples in this study, B) soils separated by carbonate and terrigenous sedimentary settings, C) soils separated by coastal environmental setting (CES) in carbonate settings, and D) soils separated by CES in terrigenous settings. In Panel A, the blue line is the linear trend calculated for this mangrove dataset and the green line is the second-order polynomial relationship for coastal marshes identified by Craft et al., (1991). For panels C and D, C_E is carbonate estuary; C_OpC is carbonate open coast; T_D is terrigenous delta; T_E is terrigenous estuary; T_L is terrigenous lagoon; and T_OpC is terrigenous open coast. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

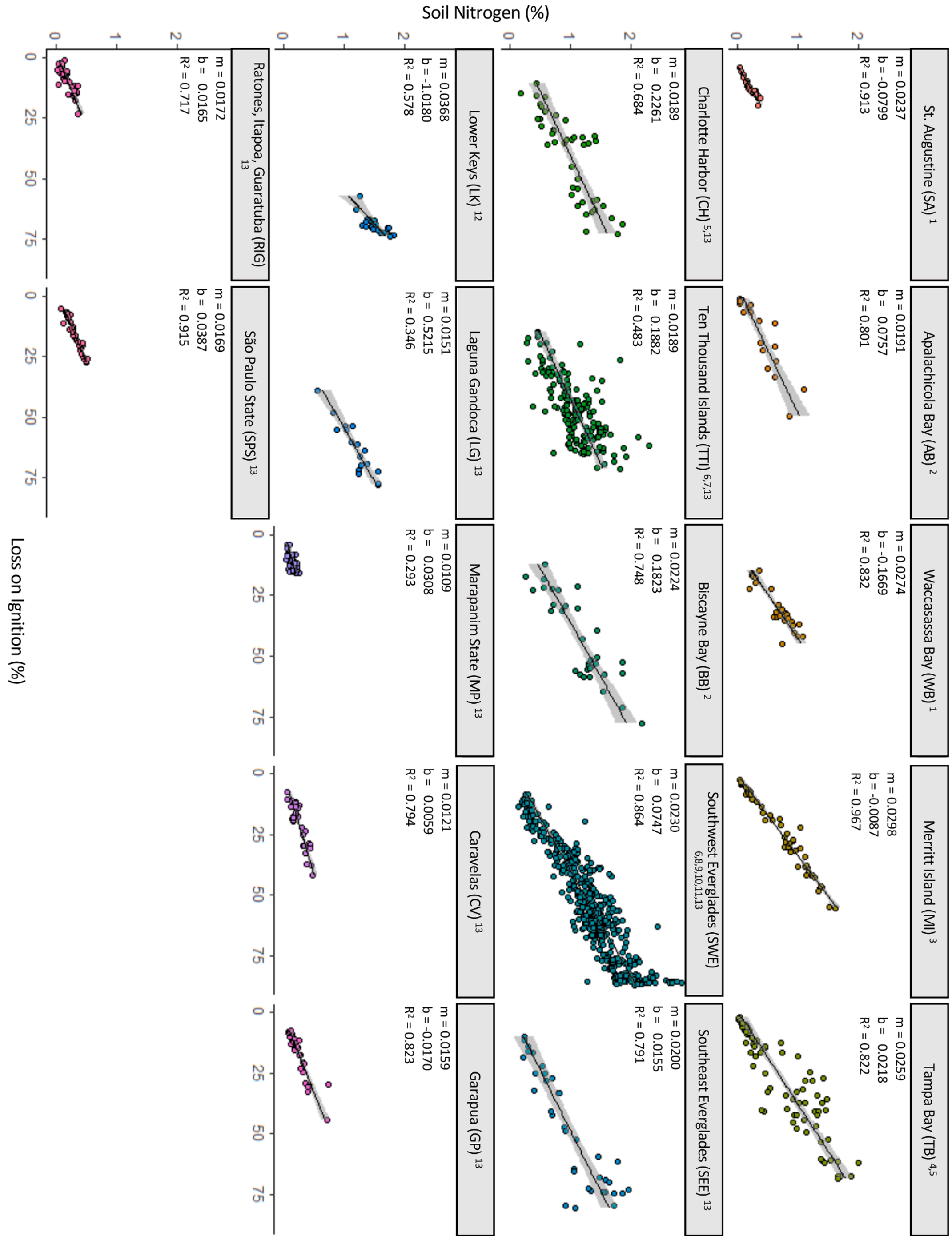

Fig. 5. Relationship between loss-on-ignition (LOI) and soil total nitrogen (%) for each region with linear trends plotted in solid black. Shaded regions denote 95% confidence interval of regression line. Superscripts following regional names correspond to the following references: [1] Vaughn et al., (2020); [2] previously unpublished; [3] Steinmuller et al., (2020); [4] Radabaugh et al., (2018); [5] Harttung et al., (2021); [6] Breithaupt et al., (2017); [7] Breithaupt et al., (2020); [8] Smoak et al., (2013); [9] Breithaupt et al., (2014); [10] Breithaupt et al., (2019); [11] Comparetto (2018); [12] Chappel (2018); [13] Rovai et al., 2018.

Highest mean TN:LOI values occurred in carbonate open coasts (0.024 ± 0.001), carbonate estuaries (0.024 ± 0.001), and terrigenous lagoons (0.024 ± 0.001), which did not differ from each other (Fig. 6).

3.4. Evaluation of performance by equation type

Of the four equation types for estimating soil TN content produced in this study, the regional equation type had the highest average R^2 of 0.73, and the lowest average RMSE of 0.14 (Table 2; Fig. 5). When applied to the 17 regional datasets, the three categorical equation types (general, sedimentary setting, and CES) were less effective on average than the regional equations. The highest average R^2 when applied to the 17 regions occurred with the CES equations (0.56), followed by the sedimentary setting equations (0.36) and the general equation (0.01). These low averages for the general and sedimentary setting equations were influenced substantially by just a few extremely poor R^2 values observed in terrigenous deltaic sites; when these were excluded the average R^2 values of the CES, sedimentary setting, and general equations were highly similar, ranging from 0.55 to 0.61 (Table 2). Average RMSE values of the three categorical equations were almost identical and did

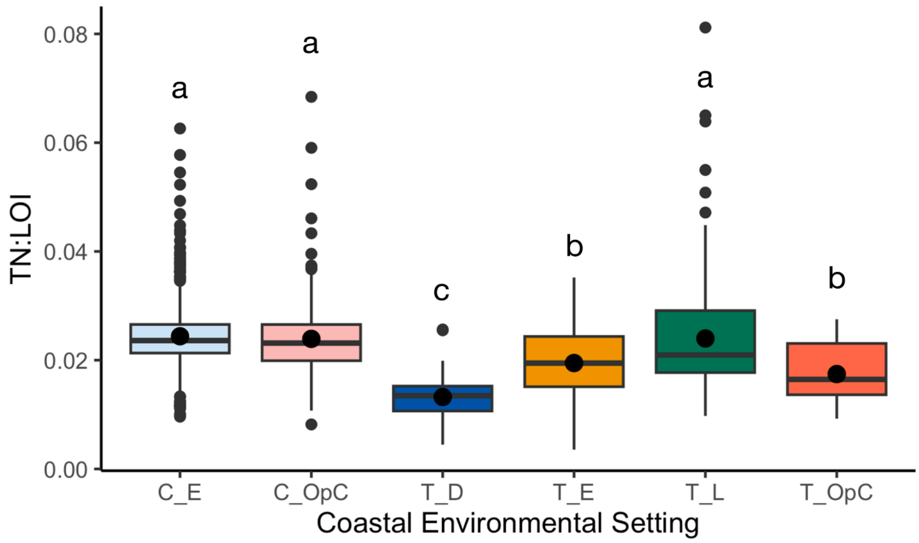

Fig. 6. Boxplots of TN:LOI for all soil core depth intervals grouped by coastal environmental setting. Horizontal lines denote the median and large circles within boxes represent the mean. Lower and upper hinges of boxes denote the first and third quartiles, with upper and lower whiskers denoting values within 1.5 standard deviations of the interquartile range. Points outside the whiskers denote outliers. Different lowercase letters indicate significantly different means ($p < 0.05$; Table S3).

Table 2
Comparison of regional conversion equation fits (adjusted for different sample sizes; Adj. R^2) and root mean square error (RMSE) with those of the general equation (Eq. 1), one of two sedimentary setting equations (Eqs. 2 and 3), and one of six coastal environmental setting (CES) equations (Eqs. 4 – 9) when fitted to the 17 regional datasets. Note that units for RMSE are TN as % of soil dry weight. Average and adjusted average (excluding negative R^2 values) are used to provide an evaluation of performance across the 17 regions.

Region	CES	Regional Equations		General Equation		Sed. Setting Equations		CES Equations	
		Adj. R^2	RMSE	Adj. R^2	RMSE	Adj. R^2	RMSE	Adj. R^2	RMSE
AB	T_L	0.80	0.13	0.75	0.15	0.71	0.16	0.76	0.14
BB	C_OC	0.75	0.22	0.66	0.26	0.70	0.24	0.66	0.38
CH	C_E	0.68	0.22	0.63	0.24	0.66	0.23	0.65	0.23
CV	T_D	0.79	0.05	−3.86	0.25	−2.13	0.20	0.79	0.05
GP	T_OC	0.82	0.06	−0.19	0.17	0.32	0.13	0.54	0.10
LG	T_L	0.35	0.37	0.20	0.41	0.23	0.41	0.22	0.41
LK	C_OC	0.58	0.11	0.05	0.16	0.05	0.16	0.44	0.22
MI	T_L	0.97	0.08	0.82	0.19	0.73	0.24	0.83	0.19
MP	T_D	0.29	0.05	−3.41	0.13	−0.94	0.08	0.29	0.05
RIG	T_E	0.72	0.06	0.44	0.08	0.61	0.07	0.71	0.06
SA	T_L	0.91	0.03	0.21	0.08	0.85	0.04	−0.07	0.10
SEE	C_E	0.79	0.24	0.67	0.30	0.65	0.30	0.64	0.32
SPS	T_L	0.92	0.03	0.38	0.09	0.76	0.06	0.25	0.10
SWE	C_E	0.86	0.20	0.86	0.24	0.86	0.20	0.86	0.20
TB	C_E	0.82	0.24	0.79	0.26	0.80	0.25	0.81	0.25
TTI	C_OC	0.48	0.25	0.45	0.26	0.44	0.26	0.43	0.30
WB	T_OC	0.83	0.11	0.77	0.12	0.81	0.11	0.79	0.12
Average		**0.73**	**0.14**	**0.01**	**0.20**	**0.36**	**0.19**	**0.56**	**0.19**
Adj. Avg.				**0.55**	**0.20**	**0.61**	**0.19**	**0.60**	**0.19**

not change with exclusion of the poor fits from terrigenous deltaic sites.

4. Discussion

4.1. Considerations when predicting TN from LOI

The equations derived from this dataset to predict TN from LOI represent the first for mangrove soils (Fig. 4, Table 1). The general equation (Eq. 1) is comparable to the quadratic equation derived for salt marsh soils by Craft et al., (1991); the goodness-of-fit of the salt marsh equation applied to our data was 0.86, essentially the same as the R^2 of 0.88 for our general equation (Eq. 1). Importantly, these data indicate that the predictive relationship also varies among CES and thus use of a single conversion equation may obscure important environmental differences. Differences among CES have been shown to predict OC:LOI ratios, soil OC stocks, soil OC burial rates, differences in forest structure and biomass, and root productivity (Rovai et al., 2018; 2021; Breithaupt and Steinmuller, 2022; Arnaud et al., 2023; Breithaupt et al., 2023). These previously observed differences have been attributed to gradients in terrigenous riverine inputs across different geomorphic types. Interestingly, the variability in the TN:LOI relationship in mangrove soils is nearly identical to that of the OC:LOI ratio (Breithaupt et al., 2023). The difference between the CES with the lowest and highest ratios varies by factors of 1.87 and 1.91 for the TN:LOI and OC:LOI ratios, respectively (Fig. 4).

We propose three primary reasons why TN:LOI varies by CES: mineral sediment dilution, biomass allocation, and variable IN content. First, ratios of TN:LOI generally increased with decreasing riverine influence, which suggests a possible dilution effect of LOI (Table 1, Fig. 6). For instance, terrigenous sedimentary settings with the highest riverine influence (deltas) displayed lower LOI means and ranges than the rest of the terrigenous sedimentary settings (Table 1, Fig. 6). These systems are characterized by high inputs of allochthonous minerogenic sediment (Worthington et al., 2020; Balke and Friess, 2016) that is incorporated into the soil matrix, effectively diluting the LOI signature (Twilley et al., 2018). In addition to sediment input, hydrological variations across different geomorphic settings also affect growing conditions by influencing nutrient availability and limitation (Twilley et al., 2018). High nutrient availability within the terrigenous deltas allows for resident mangrove species to allocate more nutrient resources to aboveground biomass (Twilley et al., 2018; Rovai et al., 2021), consequently contributing to lower relative TN allocation to root development and biomass in the soil matrix. Conversely, mangroves found in terrigenous lagoons and open coasts within sedimentary environments tend to experience greater nutrient limitation, primarily due to phosphorus deficiency. As a result, these mangroves allocate more nutrients to root growth, which typically contributes to higher TN:LOI ratios in soils (Twilley et al., 2018; Rovai et al., 2021). Estuaries were expected to follow a similar trend in TN:LOI as deltas, as both environments are characterized by allochthonous sediment inputs (Breithaupt et al., 2023). The close clustering of terrigenous estuaries with terrigenous open coasts is likely due to the small sample size of terrigenous estuaries ($n = 35$) and a narrower range of LOI values. Only one region (RIG) accounted for all terrigenous estuary observations (Table S2); more observations from terrigenous estuaries that account for a larger range of LOI would improve our ability to define the relationship of TN:LOI between terrigenous deltas, estuaries, and open coasts.

A third potential reason for differences in TN:LOI among CES may be a systematic variation in IN. In most organic soils, IN is often less than 10 % of the TN pool (Stevenson, 1982; Schulten and Schnitzer, 1997; Fig. 1), though in marine sediments IN has been observed to represent between 15 and 45 % of TN (Freudenthal et al., 2001; Zheng et al., 2015). Furthermore, IN content of TN in mangrove, salt marsh, and seagrass soils has been reported as high as 26, 44, and 32 %, respectively in cases where high nitrification rates occur (Li et al., 2023). This may suggest that CES with the highest TN:LOI that are limited by terrigenous riverine influence (carbonate settings and terrigenous lagoons, Fig. 6), have greater relative presence of IN. Although we do not have separate measurements of organic and inorganic fractions of TN for our samples, the multiple strong linear relationships with LOI (Table 1) suggest that IN is relatively constant and/or relatively scarce in these samples. This assumption that much of TN is expected to be organic allows for the practicality of relating TN to LOI. If IN was a larger fraction of TN, the relationship would likely be much more unpredictable, with greater variability and less linearity. Such an assumption is analogous to how LOI functions as a good predictor of OC, but not total C, because OC and IC are not mutually dependent and IC content can vary widely in coastal sediments. Despite the strong linearity in TN and LOI observed in our dataset, it should be noted that TN measured with an elemental analyzer is typically determined via combustion at approximately 960 °C. Thus, minimally weathered, coarse-grained sediments may not yield their rock-bound forms of IN below temperatures of 1000–1200 °C (Sadofsky and Bebout, 2000; Pinti and Hashizume, 2001). The variability of IN across these regions likely contributes to the variability within the y-intercepts of the regional linear relationships between TN and LOI (Fig. 5). It is possible that residual IN might be manifested as a positive y-intercept in a conversion equation as it would indicate the presence of IN in the absence of LOI-estimated organic matter. However, the highest y-intercepts in this dataset occurred in the equations for general carbonate settings (0.0008), carbonate estuaries (0.0007), and carbonate open coasts (0.0019; Table 1); these are not settings where terrigenous sedimentary rock weathering products, including silicate-bound NH_4^+, are found.

In general, the ratios of TN:LOI were similar within sedimentary settings, except for terrigenous lagoons which grouped with the two carbonate settings (Table S2; Fig. 6). Terrigenous lagoons have relatively reduced riverine input, with less terrestrial P inputs, higher soil N:P ratios, and thus higher biomass allocation to roots (Rovai et al., 2018). Moreover, these three settings may share common forms and sources of N deposition and fixation, or post-depositional changes related to nitrogen or bulk organic matter cycling.

Within carbonate settings, riverine influence seems to have little effect on TN:LOI. Instead, the grouping of carbonate CES together suggests that sedimentary setting might be the major predictor of TN:LOI, rather than geomorphic setting. This may indicate the important role of autochthonous abiotic (e.g., precipitation of ooids in whitings; Purkis et al., 2023) and biotic (calcareous macro-and micro-algal production; Munnecke et al., 2023) processes for the formation of carbonate muds in these settings compared to more river-derived sediments in terrigenous, siliciclastic coastal settings. Within these environments, hydrology is dominated by freshwater sheet flow (carbonate estuaries; Obeysekera et al., 1999) or interaction with the marine environment (carbonate open coasts), which relieves nutrient limitation to stimulate primary production and organic matter accumulation without diluting the soil matrix with large amounts of allochthonous minerogenic sediment.

4.2. Choosing a conversion equation

We suggest a step-based approach to evaluating the options for quantifying TN in mangrove soils (Fig. 7). The most accurate method of identifying TN concentrations in mangrove soils is by direct measurement using an EA. However, in the absence of access or funding for the use of such an instrument, our results suggest conversion equations can effectively estimate soil TN content where only LOI data are available. When limited funding is available to analyze some but not all samples in a dataset, our results indicate the creation of a regionally specific LOI-to-TN equation is the most accurate way to estimate TN, as this approach explained more of the average variance in the data than the categorical equation types and had the lowest average RMSE (Table 2). When no funding is available to directly measure TN of any samples, the first question to ask is whether the sedimentary setting is known, either by representation on a global mapping product, such as that provided by Worthington et al., (2020) or based on local expertise. If the sedimentary setting cannot be ascertained with confidence, the best estimation approach is to use the general equation (Eq. (1); Table 1). If the sedimentary setting is known but not the geomorphic setting that would identify the CES, we recommend using Eq. (2) for carbonate settings or Eq. (3) for terrigenous settings. When the CES for a given location is known, then Eqs. 4 – 9 should be utilized; after the creation of a regional equation, the use of CES equations was considered the most accurate estimation approach.

Lastly, we note that results presented here were obtained from samples where dry mass of samples was recorded following achievement of a constant dry weight, and LOI was conducted at 550 °C for three hours. Samples that are not fully dried or that are subjected to varying times or temperatures during LOI will probably exhibit a slightly different LOI-to-TN relationship. The LOI process has been demonstrated to vary substantially due to deviations in LOI procedure, soil type, and drying techniques (Ball, 1964; Dean 1974; Santisteban et al., 2004; Hoogsteen et al., 2015). Variability in temperatures and durations can result in incomplete combustion of SOM, which occurs at low temperatures (Ball, 1964) or the decomposition of soil carbonates, which occurs at higher temperatures (Kasozi et al., 2009). These differences in temperature and duration can result in under- or overestimation of SOM via the LOI process. Furthermore, incomplete drying prior to combustion can result in an overestimation of SOM, particularly if the sample has a high clay mineral content, which retain structural water during the drying process (Sun et al., 2009).

4.3. Future directions

From a climate change mitigation perspective, the 'blue carbon'

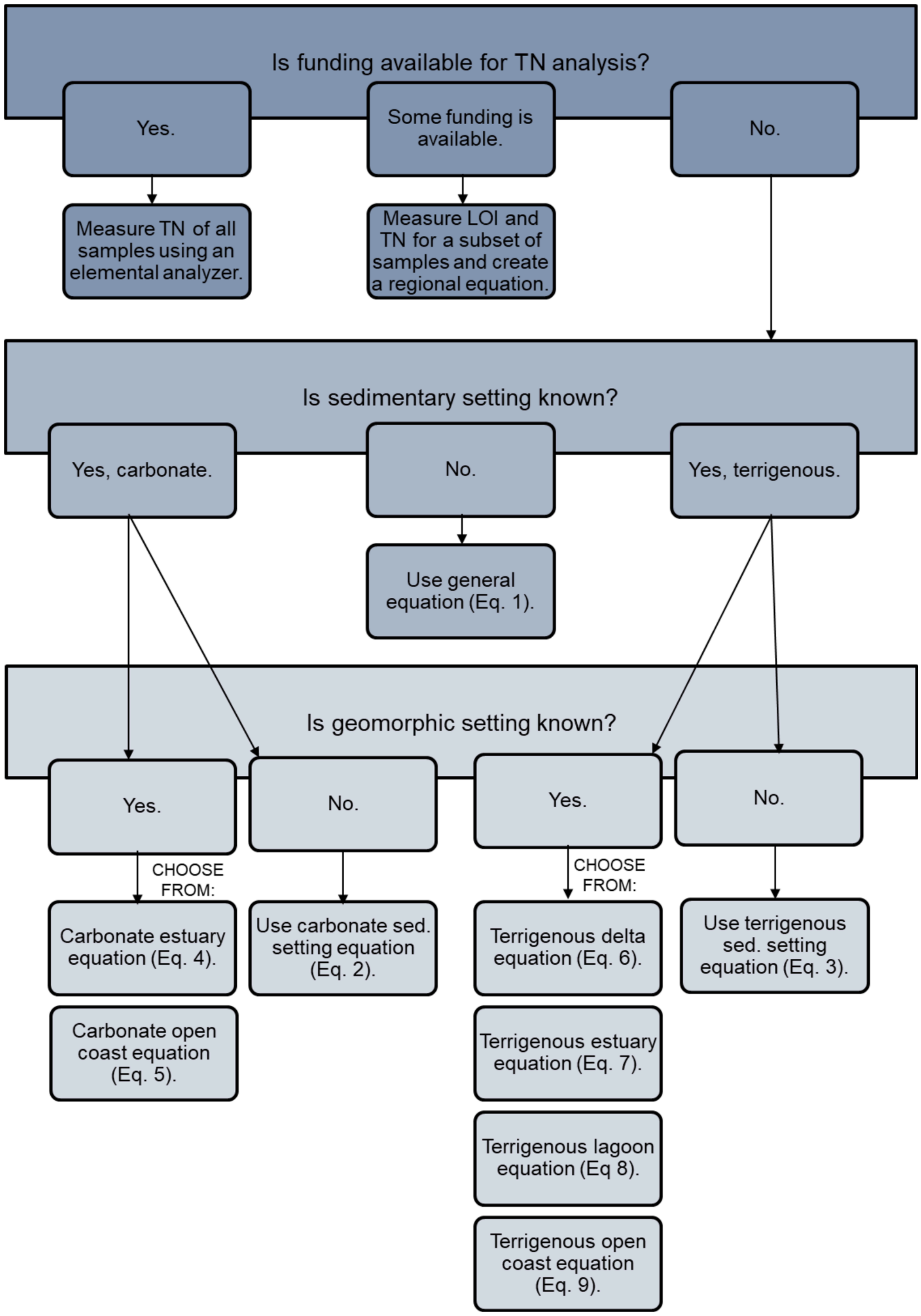

Fig. 7. Decision tree for identifying options for quantifying total nitrogen content of mangrove soils. Equation numbers are identified in Table 1.

concept catalyzed increased interest in accounting for the ability of coastal vegetated environments to extract CO_2 from the atmosphere more so than in their ability to intercept allochthonous OC. In contrast, the concept of "blue nitrogen" is likely to emphasize the importance of intercepting and retaining allochthonous N that would otherwise be transferred from the continents to the sea where it can contribute to eutrophication and its associated declines in marine ecosystem health. Much remains unknown regarding this interception function of coastal ecosystems in global N accounting. There has been growing interest in quantifying the fluxes and storage of nitrogen in mangrove forests (Reis et al., 2017a; b; Pérez et al., 2021; Alongi, 2020; Wigand et al., 2021). The application of conversion equations to relate TN to LOI, whether regionally or by CES, opens an opportunity for increased collection of coastal nitrogen data by providing a low-cost alternative to direct measurement of TN in mangrove soils. Beyond simply adding to the body of literature concerning mangrove nitrogen cycling, care should be taken to diversify research in mangrove soils to fully represent both global regions and different types of CES. This dataset represents a limited spatial extent that should be expanded. For example, there are no observations from carbonate lagoons, which are estimated to account for 5.08 % of mangroves worldwide (Worthington et al., 2020). The dataset also has relatively few observations from terrigenous deltas and estuaries (n = 72 and n = 36, respectively), though these systems are estimated to account for 39.7 % and 27.2 % of global mangroves, respectively (Worthington et al., 2020). Bolstering this dataset of 1275 observations with new observations of LOI and TN from diverse geomorphic settings supporting mangrove species across the globe would contribute to improved equations, perhaps illuminating further differences between the equations for each CES and improving our understanding of the role that mangroves play in intercepting and sequestering N along its path from land to sea.

Loss-on-ignition as a procedure for quantifying soil organic matter is widespread, has been used for nearly 100 years, and remains a recommended method for the estimation of OC content in scenarios that require a low-cost approach (Howard et al., 2014; Hoogstein et al., 2016). The data presented herein used samples that were dried until a constant weight was achieved and followed consistent combustion temperatures and durations. Some of the issues that can affect the LOI procedure, including the presence of clays and variation in combustion temperatures and times, can contribute to an over- or underestimation of SOM, (Ball, 1964; Kasozi et al., 2009; Sun et al., 2009; Hoogsteen et al., 2015). Therefore, differences from the relationships identified here may occur if users employ different LOI procedures. While we recommend that LOI be accompanied by EA analysis of soil constituents, the conversion equations provided by this work provide an alternative means for using LOI to estimate mangrove soil TN content.

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CRediT authorship contribution statement

Havalend E. Steinmuller: Writing – review & editing, Writing – original draft, Visualization, Formal analysis, Data curation, Conceptualization. **Joshua L. Breithaupt:** Writing – review & editing, Writing – original draft, Formal analysis, Conceptualization. **André S. Rovai:** Writing – review & editing. **Kevin M. Engelbert:** Writing – review & editing, Conceptualization. **Joseph M. Smoak:** Writing – review & editing. **Lisa G. Chambers:** Writing – review & editing. **Kara R. Radabaugh:** Writing – review & editing. **Ryan P. Moyer:** Writing – review & editing. **Amanda Chappel:** Writing – review & editing. **Derrick R. Vaughn:** Writing – review & editing. **Thomas S. Bianchi:** Writing – review & editing. **Robert R. Twilley:** Resources. **Paulo R. Pagliosa:** Resources. **Miguel Cifuentes-Jara:** Writing – review & editing. **Danilo Torres:** Resources.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.geoderma.2024.116956.

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