Loss on Ignition and Kjeldahl Digestion for Estimating Organic Carbon and Total Nitrogen in Estuarine Marsh Soils: Calibration with Dry Combustion[1]

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ABSTRACT: Soils (n = 250) were collected from ten salt and brackish-water marshes of North Carolina and analyzed for organic matter content by loss on ignition (LOI) and Kjeldahl nitrogen (KN). Total organic carbon and total nitrogen were determined on the same samples using an elemental CHN analyzer. Regression analyses indicated that LOI and KN were excellent estimators of organic C (R^2 = 0.990) and total N (R^2 = 0.986), respectively, in low clay content (0-11%) marsh soils containing a wide range of soil organic C (0.1-28%) and total N (0-1.6%). A quadratic equation best described the relationship between organic C and organic matter (Organic C = 0.40 [LOI] + 0.0025 [LOI]^2) while a linear model accurately described the relationship between total N and Kjeldahl N (Total N = 1.048 [KN] − 0.010). The proportion of organic C in organic matter (C/OM) increased with increasing soil organic matter content, probably as a result of aging. Young marshes, which are characterized by low soil organic content contain C/OM ratios similar to emergent vegetation (40-45%). In old organic soils (70-80% organic matter), C/OM increased to 57-60% due to accumulation of reduced organic materials.

Introduction

Marsh soils are a major reservoir of organic carbon (C) and total nitrogen (N) in estuarine ecosystems (Friedman and DeWitt 1978; Craft et al. 1988b) and are important sinks in the global C cycle (Feijtel et al. 1985; Armentano and Menges 1986). Studies of estuarine nutrient cycles and food webs often rely on the measurement of soil organic matter content by loss on ignition (LOI) (Nixon and Oviatt 1973; Lord 1980; Morris and Bowden 1986; Craft et al. 1988b) or by dichromate oxidation (Cammen 1975; Lindau and Hossner 1981; Broome et al. 1986). Both LOI and dichromate methods require correction factors for estimating soil organic C.

The amount of organic C recovered by dichromate oxidation is highly variable, and ranges from 44% to 92% of the soil organic matter (Nelson and Sommers 1982). Other disadvantages of the dichromate method include interference from chloride and ferrous ions (both of which may be abundant in reduced, estuarine marsh soils; Nelson and Sommers 1982) and the problem of safe disposal of the dichromate-sulfuric acid solution.

Loss on ignition (LOI) is a rapid, safe, and in-

expensive method for measuring soil organic matter. The presence of carbonates does not affect the magnitude of the ignition loss (Davies 1974). However, loss of structural water during ignition may overestimate organic matter content in soils containing large amounts of clay (Mook and Hoskin 1982).

Factors ranging from 0.22 to 0.52 have been used to covert organic matter to organic C in marsh soils (Lord 1980; Nixon 1980; Bowden 1984; Morris and Whiting 1986: Craft et al. 1988b). This wide range suggests that, in many studies, LOI may yield inaccurate estimates of organic C.

The Kjeldahl procedure is widely used for estimating total N (KN) in marsh soils (Lindau and Hossner 1981; Bowden 1984; Morris and Bowden 1986; Craft et al. 1988b). The standard Kjeldahl procedure recovers ammonium and much of the organic N. Without appropriate modifications, however, it does not quantitatively recover nitrogen in compounds containing N-O or N-N linkages, or nitrogen in refractory heterocyclic compounds (Bremner and Mulvaney 1982).

We have compared LOI and KN with the dry combustion technique for measuring organic C and total N. Ten estuarine marshes containing a wide range of soil organic C and total N were used. Our goal was to generate regression equations for converting soil organic matter as determined by LOI to organic C and Kjeldahl N to total N.

Methods

Soils were collected from ten estuarine marshes in eastern North Carolina. The sites included both mineral (Carteret series; mixed, thermic typic psammaquents) and organic (Lafitte series; euic, thermic typic medisaprists) soils (Daniels et al. 1984). Soil organic matter content ranged from 0% to 72%. A detailed site description is presented by Craft et al. (1988b). Soils were sampled by taking 10 to 20 cores (8.5 cm diameter by 30 cm deep) from each marsh. Cores were subdivided into 0–10 and 10–30 cm sections and the soil was air-dried, ground, and passed through a 2 mm-mesh diameter sieve.

Samples were tested for carbonates by treating with 6 N HCl and observing effervescence. Carbonates were present in soils collected from two of the ten marshes. Soils taken from these two marshes were treated with 6 N HCl before analyzing for organic C. The clay content of the marsh soils ranged from 0% to 11% (Craft et al. 1986; Sacco 1989). Soils containing appreciable amounts (6–11%) of clay also had high organic matter levels (19–51%) (Craft et al. 1986; Sacco 1989). Mook and Hoskin (1982) observed that an estuarine sed-

iment containing 87% clay, 13% silt, and 0% organic matter lost 10% of its mass after ignition at 450°C. Based on this estimate, we calculate that structural water accounts for only 2% of the ignition loss in our high clay content (11%) soil. For this reason, we believe the contribution of structural water to ignition loss in our soils was negligible.

Two hundred and fifty samples were analyzed for organic C and total N. The LOI and Kjeldahl N data were published previously by Craft et al. (1988b). Organic matter content was measured on weighed 5 cm or 10 cm³ samples by loss on ignition at 450°C for 8 h. Kjeldahl N was determined on 0.25–1.0 g soil samples according to Bremner and Mulvaney (1982). Organic C and total N were measured on 5–75 mg samples using a Perkin-Elmer 2400 CHN analyzer (Nelson and Sommers 1982). Organic matter, organic C, total N, and Kjeldahl N values were corrected to oven-dried weight by drying a subsample at 105°C overnight.

The CHN analyzer was calibrated using acetanilide (71.09% C, 10.36% N). Carbon and nitrogen values for acetanilide standards (n = 17) were 70.99 ± 0.07% (± 1 standard error) and 10.36 ± 0.04%, respectively. Analysis of soybean leaf (nitrogen = 4.32%) standards (n = 20) yielded values of 43.66 ± 0.04% for C and 4.33 ± 0.02% for N.

Fifty soil samples were randomly selected for duplicate analysis. Mean coefficients of variation (CV) were 15.75% and 6.00% for LOI and organic C, respectively. Likewise, the dry combustion technique for total N (mean CV = 9.80%) was more precise than the Kjeldahl method (mean CV = 11.15%).

Regression analysis was used to describe the relationships between the dependent variables, organic C and total N, and the independent variables, Kjeldahl N and organic matter. The data were square-root transformed to improve homogeneity of variance. Residuals were plotted against the independent variable to determine whether a linear or quadratic equation was more appropriate. Once a general model was chosen, the best model (regression through the origin versus simple regression) was determined by an F test (a = 0.05) of the difference in the sum of squares error (SSE) and degrees of freedom of the error term (df) between the reduced and full models according to the expression (Steel and Torrie 1980):

$$\frac{\text{SSE (reduced model)} - \text{SSE (full model)}}{\text{df (reduced model)} - \text{df (full model)}} \Big/ \begin{array}{c} \text{Mean Square Error} \\ \text{(full model)} \end{array}$$
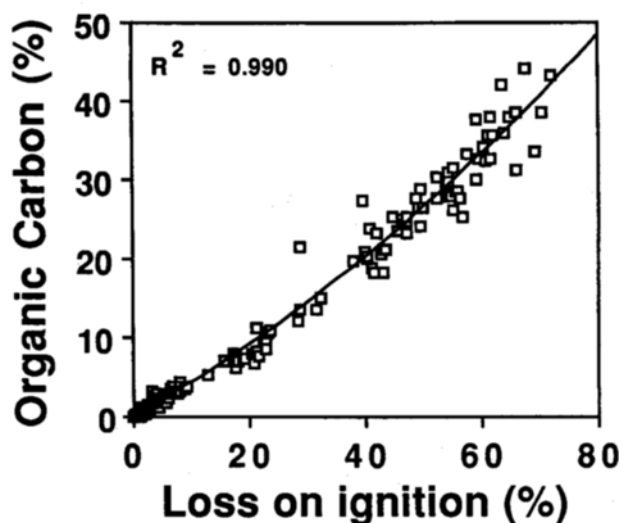
Fig. 1. The relationship between organic carbon and loss on ignition (LOI) in marsh soils (n = 250). The regression equation (± 1 standard error) is Organic C = (0.40 ± 0.01)LOI + (0.0025 ± 0.0003)LOI².

Fig. 2. The relationship between total nitrogen and Kjeldahl nitrogen (KN) in marsh soils (n = 250). The regression equation (± 1 standard error) is Total N = (1.048 ± 0.008)KN − (0.010 ± 0.005).

Results and Discussion

REGRESSION ANALYSES

The relationship between organic C and organic matter was described by the equation:

$$\text{Organic C} = (0.40 \pm 0.01)\ \text{LOI} + (0.0025 \pm 0.0003)\ \text{LOI}^2.$$

The quadratic form (Fig. 1) suggests that the proportion of organic C in soil organic matter (C/ OM) increases with soil organic matter content. This does not appear to be due to different organic C sources. Estuarine marshes accumulate soil organic matter by deposition of residues of net primary production (NPP) from emergent vegetation (Craft et al. 1988a). Because marsh emergents such
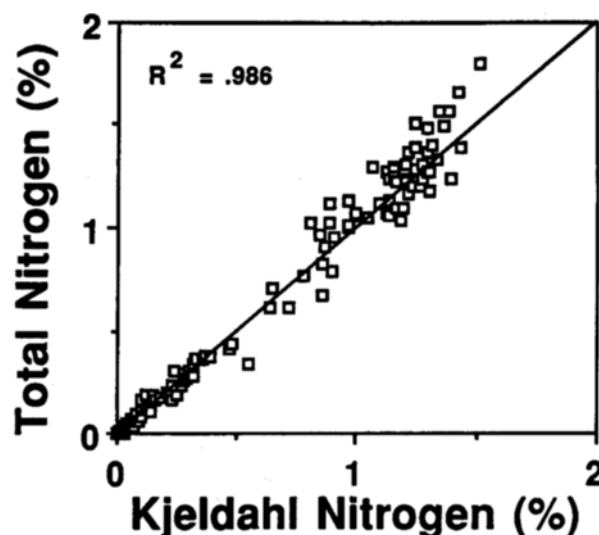
TABLE 1. Factors for converting soil organic matter to organic carbon in wetland and upland soils.

	Reference	Conversion Factor
Wetland:		
(marsh)	Morris and Whiting (1986)[a]	0.225
(marsh)	Craft et al. (this study)[a]	0.400–0.600
Upland:		
(litter)	Lunt (1931)[a,b]	0.529–0.556
	Goldin (1987)[a]	0.417
	David (1988)[a]	0.51–0.53
(mineral soil)	Goldin (1987)[a]	0.405
	David (1988)[a]	0.520
(litter and	Ball (1964)[b]	0.458–0.476
mineral soil)	Ranney (1969)[a]	0.556

[a] Calibrated by dry combustion.
[b] Calibrated by chromic acid oxidation.

as *Spartina, Juncus,* and *Cladium* have similar tissue organic C content (42–45%) (Craft et al. 1986), different C sources do not explain the increase in C/OM with increasing soil organic matter content.

Differences in the ages of the marshes could account for the increase in C/OM with increasing soil organic content. Young marshes are characterized by low soil organic content (Craft et al. 1988b). The organic matter accumulating in these young soils is recently deposited plant material and the C/OM values reflect those of emergent vegetation. As marshes age, the interaction of water-saturated soils and deposition of NPP result in continued accumulation of soil organic matter. Over time, anaerobic decomposition produces reduced compounds, such as refractory organic compounds, fatty acids, and methane (Stevenson 1986; Reddy and Graetz 1988), containing a higher C content than "fresh" plant tissue. Thus, C/OM increases with increasing soil organic matter content (and marsh age) through accumulation of reduced organic compounds.

With the exception of Morris and Whiting (1986), published conversion factors for estimating organic C from LOI data in wetland and upland soils are in close agreement (Table 1). High clay content or the presence of carbonates in their low organic C (1–4%) soils could explain the low conversion factor used by these researchers.

Regression analysis indicated that the Kjeldahl method recovered approximately 95% of the total soil N (Fig. 2). It is likely that the shortfall is in the
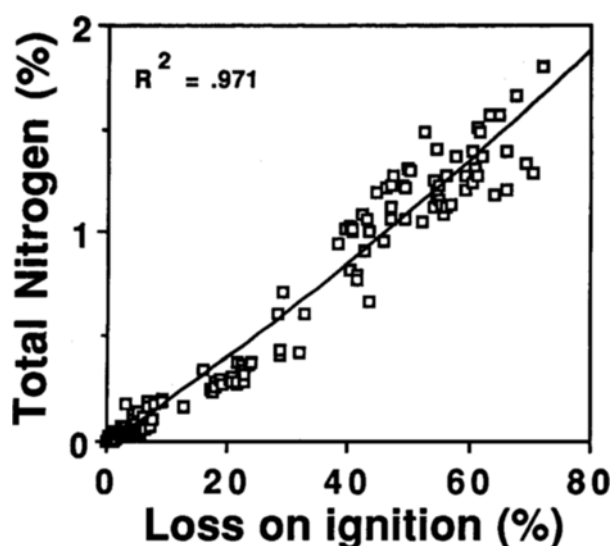
Fig. 3. The relationship between total nitrogen and loss on ignition (LOI) in marsh soils (n = 250). The regression equation (± 1 standard error) is Total N = (0.020 ± 0.001)LOI + (0.00005 ± 0.00002)LOI^2 − (0.018 ± 0.008).

form of refractory heterocyclic compounds and compounds containing N-N and N-O linkages (Bremner and Mulvaney 1982).

The relationship between total N and organic matter was best described by the quadratic equation:

$$\text{Total N} = (0.020 \pm 0.001)\text{LOI} + (0.00005 \pm 0.00002)\text{LOI}^2 - (0.018 \pm 0.008)$$

The proportion of N in organic matter (N/OM) increased with increasing soil organic content (Fig. 3). The large coefficient of determination (R^2 = 0.971, n = 250) for this model indicates that soil organic matter content as measured by LOI is useful also for estimating total N in marsh soils.

MARSH SOIL ORGANIC CARBON AND TOTAL NITROGEN

Marsh soil organic C, organic matter content (LOI), and total N ranged from 0.11% to 28.09%, 0.2% to 51%, and 0% to 1.63%, respectively (Table 2). Three of the marshes (N.C. Phosphate #2, Snow's cut #2, Texasgulf #2) were underlain by organic soils (Typic Medisaprists). Percent organic C was greater in the 10–30 cm depth compared to the 0–10 cm depth in these three marshes (Table 2). Likewise, total N increased with depth in two of the three organic soils.

Carbonates were present in soils collected from the Pine Knoll Shores marshes (Table 2) and accounted for 10–31% and 29–45% of the total C

TABLE 2. Mean organic carbon, loss on ignition (LOI), total nitrogen and elemental organic carbon : nitrogen (C:N) ratios in the ten marshes. (Values in parentheses are the number of samples collected from each depth within a given marsh).

Site	Depth (cm)	Organic C (%)	LOI[a] (%)	Total N (%)	C:N (wt : wt)
N.C. Phosphate #1	0–10	0.21	1.1	0.01	24.5
(15)	10–30	0.17	1.0	0.01	19.8
N.C. Phosphate #2	0–10	24.92	48.0	1.63	17.8
(20)	10–30	27.50	50.9	1.52	21.1
Oregon inlet #1	0–10	1.40	2.4	0.10	16.3
(10)	10–30	0.11	0.2	0	—
Oregon inlet #2	0–10	2.46	5.5	0.18	15.9
(10)	10–30	0.60	1.6	0.03	23.3
Pine Knoll Shores #1	0–10	0.29 [0.13][b]	1.2	0.02	16.9
(10)	10–30	0.11 [0.09]	0.4	0	—
Pine Knoll Shores #2	0–10	0.36 [0.04]	1.2	0.03	14.0
(10)	10–30	0.20 [0.08]	0.6	0.01	23.3
Snow's cut #1	0–10	1.42	3.9	0.08	20.7
(10)	10–30	0.27	1.2	0.01	31.5
Snow's cut #2	0–10	7.41	19.3	0.37	23.4
(10)	10–30	10.60	23.5	0.51	24.2
Texasgulf #1	0–10	2.22	5.0	0.07	37.0
(15)	10–30	1.89	3.8	0.05	44.1
Texasgulf #2	0–10	22.79	44.0	1.32	20.1
(15)	10–30	28.09	48.9	1.52	21.6

[a] From Craft et al. 1988b.
[b] Percent carbonate-carbon.

present in the 0–10 and 10–30 cm depths, respectively.

Elemental organic C:N ratios ranged from 14.0 to 44.1 (Table 2). In nine of the marshes, the C:N ratios indicated net mineralization (C:N < 20) of organic N or an equilibrium (C:N = 20–30) between mineralization and immobilization (Stevenson 1986). The high C:N ratios of the Texasgulf #2 marsh suggested microbial immobilization (C:N > 30) of N in the soil of this marsh (Stevenson 1986). Organic C:N ratios generally increased from the 0–10 cm depth to the 10–30 cm depth (Table 2).

In summary, LOI is a reliable predictor of organic C and Kjeldahl N is a reliable predictor of total N in low clay content (0–11%) marsh soils containing a wide range of soil organic C (0.11–28.09%) and total N (0–1.63%). The amount of organic C in organic matter (C/OM) increases with increasing soil organic matter content. In young, low (1%) organic matter soils, C/OM is similar to marsh emergent vegetation (C/OM = 40%). Accumulation of reduced organic C compounds in

old, organic (60–80% organic matter) marsh soils produces a C/OM of 55–60%.

ACKNOWLEDGMENTS

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