Organic carbon, organic matter and bulk density relationships in boreal forest soils

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Périé, C. and Ouimet, R. 2008. **Organic carbon, organic matter and bulk density relationships in boreal forest soils**. Can. J. Soil Sci. **88**: 315–325. Relationships between soil organic carbon (SOC), organic matter (SOM), and bulk density (BD) were established in acidic loamy to sandy loam fine fractions of forest soils in Quebec (Canada). The interest of such relationships rests with the possibility of using simple and rapid techniques to estimate SOC and BD. It is also a crucial step in establishing the correspondence among several data bases when SOC data are obtained using different measurement techniques. In this study, SOC was measured by dry combustion (SOC_{DC}) and wet digestion (SOC_{WD}) methods, and organic matter by loss-on-ignition (LOI). Our results suggest that, in these soils: (1) LOI can be used for estimating SOC ($r^2 = 0.95$, RMSEP = 16%) and SOC_{DC}/SOM significantly decreased with increasing depth from 0.49 to 0.27; (2) SOC_{DC} and SOC_{WD} were highly correlated. Even if SOC_{WD} provided near complete recovery of SOC_{DC}, dry combustion remains the preferred method for SOC analysis since SOC_{WD} recovery decreased with increasing depth from 100 to 83%. (3) BD was also strongly related to SOM ($r^2 = 0.81$). We recommend using the organic density approach to estimate BD from SOM because it allows BD to be predicted without significant bias and with a degree of accuracy of 14%.

Key words: Forest soils, soil organic carbon, soil organic matter, soil bulk density

Périé, C. et Ouimet, R. 2008. **Liens entre le carbone organique, la matière organique et la masse volumique apparente dans le sol des forêts boréales**. Can. J. Soil Sci. **88**: 315–325. Les auteurs ont précisé les liens entre le carbone organique (COS), la matière organique (MOS) et la masse volumique apparente (MVA) du sol dans les fractions fines de loam et de loam sablonneux des sols forestiers acides du Québec. De tels liens présentent un certain intérêt, car on pourrait y recourir comme technique simple et rapide pour évaluer la concentration de COS et la MVA. Il s'agit aussi d'une étape cruciale quand on souhaite établir une correspondance entre des bases de données où la concentration de COS émane de diverses techniques. Dans le cadre de la présente étude, la quantité de COS a été déterminée par combustion sèche (COS_{CS}) et digestion par voie humide (COS_{DH}) tandis qu'on a mesuré la concentration de matière organique au moyen des pertes par calcination (PPC). Les auteurs ont obtenu les résultats que voici pour les sols concernés. (1) La méthode PPC permet d'estimer le COS (r^2 = 0,95, RMSEP = 16 %) et le ratio COS_{CS}/MOS diminue de manière significative, de 0,49 à 0,27, avec la profondeur. (2) Il existe une importante corrélation entre le COS_{CD} et le COS_{DH}. Bien que la technique COS_{DH} permette de récupérer la quasi totalité du COS_{CS}, la combustion sèche demeure la méthode privilégiée pour analyser le COS, la quantité de carbone récupérée par COS_{DH} passant de 100 à 83 % à mesure qu'augmente la profondeur. (3) La MVA présente également d'étroites relations avec la MOS (r^2 = 0,81). Les auteurs préconisent l'usage de la densité de la matière organique pour estimer la MVA à partir de la MOS, car cette technique n'engendre aucun biais important et sa précision atteint 14 %.

Mots clés: Sols forestiers, carbone organique du sol, matière organique du sol, masse volumique apparente du sol

Recently, our perspective about soil organic carbon (SOC) has changed. In the past, SOC concentration was primarily measured to evaluate soil "quality" (Gregorich et al. 1994; Larson and Pierce 1994; Périé and Munson 2000; Shukla et al. 2006). Presently, carbon (C) storage is also studied, in the context of greenhouse gas balance assessment, and how soil C dynamics exerts its influence on the global C cycle (Tremblay et al. 2006; Ouimet et al. 2007). The potential of using SOC both as an indicator of soil quality and a broader indicator of ecosystem response to environmental changes has reinforced the importance of having appropriate techniques to accurately measure SOC concentrations and to adequately predict C storage in soils. The choice of methodology for C assessment is critical to the accurate quantification of SOC concentration, content and change over time.

A variety of methods are available for the analysis of SOC in forest soils, each having advantages and disadvantages in terms of accuracy, expense, and convenience. For example, elemental C analyzers are

Abbreviations: **BD**, bulk density; **C**, carbon; **LOI**, loss-on-ignition; **SOC**, soil organic carbon; **SOC_{DC}**, SOC measured by dry combustion; **SOC_{WD}**, SOC measured by wet digestion; **SOM**, soil organic matter accurate, but they may be expensive to purchase, operate and maintain. In comparison, the Walkley-Black procedure (Nelson and Sommers 1982) is inexpensive and quick to perform, but exhibits variable SOC recovery and generates hazardous products such as Cr. Loss-on-ignition (LOI), which involves combusting samples at high temperatures and measuring weight loss, has been proposed as an inexpensive, convenient, and accurate method for estimating SOC (Ball 1964; Lowther et al. 1990; Konen 2002); however, an alternate view is that LOI should only be used for rough estimates of SOC (Abella and Zimmer 2007). Detailed descriptions and comparisons of these methods are documented in the literature (Nelson and Sommers 1982; Tabatabai 1996; Sollins et al. 1999). Two general approaches are currently available to determine soil organic carbon concentration: (1) wet digestion methods, involving acid hydrolysis of the organic material in the presence of excess oxidant followed by determination of unreduced oxidant via a redox titration (Walkley and Black 1934; Nelson and Sommers 1982); and (2) dry combustion in which SOC is quantitatively oxidized to, and determined as, CO_2. Since only dry combustion methods render unambiguous estimates of released C (wet digestion methods usually involve the use of correction factors), it has been recognized as a standard technique. Because many laboratories have acquired automated total C instruments in recent years, there has been a shift away from the more traditional wet oxidation procedure and its associated limitations (i.e., the need for an empirical factor to convert to total organic carbon due to incomplete oxidation, toxic wastes, interferences from chloride, Fe^{2+}, and MnO_2) to the dry combustion method (De Vos et al. 2007). Establishment of the relationship between SOC measured by dry combustion (SOC_{DC}) and wet digestion (SOC_{WD}) is a crucial step for establishing the correspondences among data bases given that SOC data are obtained using different measurement techniques.

Another indirect approach of measuring SOC is the determination of SOM by LOI. Conversions between values of organic C and organic matter are often made using a general conversion factor. For many years the Van Bemmelen factor of 1.724 was used on the assumption that, on average, SOM contains 58% of organic C. However, there is no evidence that a single value of the Van Bemmelen factor is appropriate for all soils because the SOC/SOM ratio varies depending on soil type (Jain et al. 1997) and soil depth (Westman et al. 2006).

Relationships among SOC, SOM and BD are frequently used to estimate soil C pools (Post et al. 1982). Values of BD are necessary to convert laboratory measurements of soil nutrient concentrations from a mass basis to a volume or area basis. In theory, BD is simple to estimate (oven-dry weight/total volume) but, in practice, methods to measure BD are labor intensive and time-consuming. This is especially true in low-density and root-filled organic horizons as well as in stony mineral horizons of forest soils. Fortunately, BD is closely related to SOM, which can be easily determined by LOI. BD tends to decrease as SOM concentration increases (Curtis and Post 1964). In 1970, Jeffrey suggested that the relationship between SOM and BD might be universal. Curtis and Post (1964) developed an empirical regression of log(BD) on log(SOM) for forest soils in Vermont (USA), Federer (1983) and Huntington et al. (1989) for forest soils in central New Hampshire (USA). More recently, Prévost (2004) obtained reasonably good estimates of BD by using logarithmic relationships for some forest soils in Quebec (Canada). However, Federer et al. (1993) proposed a new theoretical expression based on the organic density concept to relate BD and SOM. Organic density is the organic mass per unit soil volume. In the present paper, the logarithmic approach and the organic density concept were tested and validated. The objectives of the present study were to establish, for loamy to sandy loam soils sampled in the boreal forest of northeast Canada, relationships between: (1) SOC_{DC}, SOM and sampling depth; (2) SOC_{DC}, SOC_{WD} and sampling depth; and (3) BD and SOM.

MATERIALS AND METHODS

Soil Sampling and Analyzes

Most of the data used in this study come from soils sampled within the context of long-term soil quality monitoring conducted by the Quebec Forest Ecosystem Research and Monitoring Network (Réseau d'étude et de surveillance des écosystèmes forestiers; RESEF). Recently (2000), the procedure for sampling the mineral soil in the spatiotemporal monitoring program was modified to sample by depth increments, as proposed by Baert et al. (1998), and Palmer et al. (2002). Because of the high spatial variability of mineral horizon thickness in forest soils, and the difficulty associated with the identification of horizon boundaries, sampling spatiotemporal designs by fixed depth seemed to be more recommended than sampling by genetic horizons.

Principal characteristics of the study sites are provided in Table 1. Briefly, the 14 sites (one pedon per site) were located in the six different bioclimatic domains of the Quebec commercial forest. The soils were deep, well-drained loamy to sandy loams, classified as Ferro-Humic Podzols ($n = 1$), Dystric Brunisols ($n = 2$) or Humo-Ferric Podzols ($n = 11$) according to the Canadian System of Soil Classification (Soil Classification Working Group, 1998) and Dystric Cambisols or Orthic Podzols according to the FAO system (FAO 2000).

Two different data bases were used in this study, depending on the relationships of interest. The first data base contained mineral soil samples taken from 14 different soil profiles and was used to build the model of the relationship between SOC_{DC} and SOM ($n = 122$) and to build ($n = 125$) and validate ($n = 32$) the model of

Table 1. Some characteristics of the selected sites

		Soil taxonomy				Diagnostic B horizon characteristics						
Code[z]	Bioclimatic domain[y]	Canadian system[x]	WRB(FAO) system[w]	Deposit	Humus type	Depth (cm)	Thickness (cm)	pH	CF[v] (%)	$\mathrm{SOC}_{\mathrm{DC}}$ (%)	SOM(%)	Texture[u]
204†	6	O.HFP	OP	Glaciofluvial	Mor	7	8	5.25	32	4.3	8.4	SL
402	3	O.HFP	OP	Glacial till	Mor	1	10	5.08	8	11.6	23.1	SL
1201	3	O.HFP	OP	Glacial till	Mor	7	16	4.81	8	3.4	9.1	L
1202†	3	O.HFP	OP	Glacial till	Mor	2	6	4.27	18	4.0	9.7	SL
1203	3	GL.HFP	OP	Glacial till	Mor	3	8	4.04	18	9.4	19.7	SL
1502	3	O.HFP	OP	Glacial till	Moder	15	15	5.04	1	9.4	4.8	SiL
501	2	O.DYB	DC	Glacial till	Mor	3	14	5.45	17	2.1	5.8	SL
702	1	O.DYB	DC	Glacial till	Mull	18	18	5.49	1	3.3	5.4	SL
103	4	O.HFP	OP	Glacial till	Mor	4	15	5.15	9	1.9	6.2	L
301†	3	O.HFP	OP	Glacial till	Moder	4	5	4.8	6	8.7	18.2	L
303†	5	O.FHP	OP	Glacial till	Mor	12	15	4.99	4	6.6	13.9	SL
RF	5	O.HFP	OP	Glacial till	Mor	6	3	4.86	11	10.2	27.0	SL
GA	5	O.HFP	OP	Glacial till	Mor	5	15	4.76	7	4.9	8.4	SL
AB	5	O.HFP	OP	Glacial till	Mor	1	10	4.68	5	3.0	9.4	L

[z] Codes with this symbol, †, indicate sites where the geostatistical designs to follow temporal changes of soil characteristics were installed and where data were used: (1) to validate model built from soil profile data to establish the relationship between $\mathrm{SOC}_{\mathrm{DC}}$ and SOM, and (2) to build and validate the relationship between $\mathrm{SOC}_{\mathrm{DC}}$ and SOC determined by wet digestion ($\mathrm{SOC}_{\mathrm{WD}}$). Three sites (RF, GA and AB) did not belong to the Quebec Forest Ecosystem Research and Monitoring Network. All soils, regardless of site, were sampled using exactly the same protocol

[y] 1 = sugar maple – bitternut hickory; 2 = sugar maple – basswood; 3 = sugar maple – yellow birch; 4 = balsam fir – yellow birch; 5 = balsam fir – white birch; 6 = black spruce – moss.

[x] GL.HFP = Gleyed Humo-Ferric Podzol; O.DYB = Orthic Dystric Brunisol; O.FHP = Orthic Ferro-Humic Podzol; O.HFP = Orthic Humo-Ferric Podzol (Soil Classification Working Group 1998).

[w] DC = Dystric Cambisol; OP = Orthic Podzol (FAO 2000).

[v] CF = coase fragment (2–50 mm) volume as a percentage of whole-soil volume (130 cm^3).

[u] L = loam, SiL = silt loam, SL = sandy loam.

the relationship between BD and SOM. To capture the change in soil composition with depth, and to avoid subjective soil horizon delimitations, soils were sampled vertically in 5 cm-deep layers on one face of the 1-m-deep pedon. A detailed description of each soil profile was made and the diagnostic B horizon was sampled. In each soil profile, approximately 10 samples (130 cm^3 each) were also taken with a volumetric hammer core sampler to measure BD. The second data base had 1139 soil samples, taken from four different RESEF sites (Table 1). In these sites, the sampling plot (20 m by 20 m) contained 60 sampling points and was installed to study the temporal changes of soil characteristics (Périé et al. 2003). At each point, soil depth strata (0- to 5-cm, 5- to 10-cm, 10- to 20-cm, and at 20-cm increments, thereafter, to a depth of 100 cm or until bedrock was reached) were sampled with an auger. This data base was used to validate the model of the relationship between SOC$_{\mathrm{DC}}$ and SOM ($n = 1139$), developed with the first data base, and to build ($n = 910$) and validate (n = 229) a model of the relationship between SOC$_{\mathrm{DC}}$ and SOC$_{\mathrm{WD}}$.

All of the soil samples were frozen before processing. Soils were air dried (≥ 48 h) and sieved through a 2-mm mesh to extract the fine soil fraction for analysis. The volumetric samples were used for determination of the total and coarse fragment (2–50 mm) weights. Coarse fragment volume was calculated using the density of stones sampled at each pit. Stone density varied between 2.43 and 2.80 g cm^{-3}, and averaged 2.62 g cm^{-3}.

LOI analyses were run using a muffle furnace (model Sybron Thermoline FA1730). Equivalent volumes (approximately 8 g) of air-dry <2 mm soil fraction were placed into 30-mL crucibles. They were oven-dried at 105°C for 2 h, cooled in a desiccator, and weighed. The samples were then ashed at 550 ± 10°C in the muffle furnace overnight. After the combustion period, samples were cooled in a desiccator and weighed. SOM (g kg^{-1}) was calculated as the mass difference between oven-dry (105°C constant weight) and ashed weights. Dichromate-oxidizable organic carbon (SOC$_{\mathrm{WD}}$) was determined on <2-mm subsamples (0.250 g) oven-dried at 105°C, using the Walkley and Black (1934) method. SOC$_{\mathrm{DC}}$ concentrations were measured with an automated dry combustion instrument (model LECO CR-412). The air-dry <2-mm fraction of the soil samples was ground to pass through a 0.5-mm sieve. Subsamples (about 0.250 g) were oven-dried at 105°C for 2 h, cooled in a desiccator, and weighed before being heated to 1350°C under an oxygen atmosphere. Carbon was converted to carbon dioxide, which was measured using an infrared detection system. Total C was presumed to equal organic C since total inorganic carbon was seldom present or negligible in the non-calcareous samples (pH$_{\mathrm{water}} < 5.5$). pH was determined using a glass electrode (soil:water vol/vol = 1:2.5). SOC$_{\mathrm{DC}}$, SOC$_{\mathrm{WD}}$ and SOM concentrations were expressed in g kg^{-1} on an oven-dry (105°C) basis, whereas BD was expressed in g cm^{-3}.

Statistical Analyses

All statistical analyses were performed using SAS version 9.1.3 software (SAS Institute, Inc. 2003). Homogeneity of variance and normality of the distribution of all dependant data were verified prior to analysis and data were natural log transformed when necessary.

Relationship Between SOC$_{DC}$ and SOM

The MIXED procedure was used to study the relationship between SOC$_{\mathrm{DC}}$ and SOM so that the correlation between samples from the same pedon could be taken into account. Because the number of observations for the deeper strata was too small, we only used the first 50 cm of mineral soil depth to establish this relationship. Three different predictive models, 1, 2 and 3, were investigated:

$$SOC_{DC} = 0.58 \times SOM \tag{1}$$

$$SOC_{DC} = \beta_0 + \beta_1 \times SOM \tag{2}$$

$$SOC_{DC} = \beta_{0_{depth}} + \beta_{1_{depth}} \times SOM \tag{3}$$

where SOC_{DC} is the soil organic carbon concentration determined by dry combustion and SOM is soil organic matter concentration. The first model uses the van Bemmelen value of 0.58, which is the value generally used to convert SOM to SOC. The second model is similar to 1, but rather than having a slope value of 0.58, the slope value (β_1) is adjusted to our data and an intercept (β_0) is added if necessary. The third model, 3, permits both the intercept ($\beta_{0_{depth}}$) and the slope parameter ($\beta_{1_{depth}}$) to change with the sampling depth. In this model, the sampling depth was considered to be an ordinal variable.

Relationship Between SOC$_{DC}$ and SOC$_{WD}$

As for the relationship between SOC$_{\mathrm{DC}}$ and SOM, the MIXED procedure was used to study the relationship among SOC$_{\mathrm{DC}}$ and SOC$_{\mathrm{WD}}$ and sampling depth.

$$SOC_{DC} = \beta_0 + \beta_{depth} \times SOC_{WD} \tag{4}$$

where SOC_{DC} is the soil organic carbon concentration determined by dry combustion, SOC_{WD} is the soil organic carbon concentration determined by wet digestion, β_0 is the intercept and β_{depth} is the slope parameter (sampling depth was considered to be an ordinal variable).

Relationships Between SOM and BD

To take into account the correlation between samples from the same soil profile, mixed models were used, with the MIXED procedure of SAS for polynomial models (5) and the NLINMIX macro of SAS for the nonlinear models (6). Sampling site was considered to be a random effect in both cases:

$$Y = a + b \times SOM + c \times \log SOM + d \times (\log SOM)^2 \quad (5)$$

where SOM is soil organic matter concentration, and a, b, c and d are the regression coefficients of the best fit model (5). The response variable Y, representing BD, was analyzed with or without logarithmic transformation.

To estimate BD, a nonlinear model was also tested, according to the organic density concept developed by Federer et al. (1993):

$$BD = \frac{D_{bm} \times D_{bo}}{(SOM \times D_{bm}) + (1 - SOM) \times D_{bo}} \quad (6)$$

where BD is the soil bulk density, D_{bm} is the empirically determined bulk density of the "pure" mineral fraction, D_{bo} is the empirically determined bulk density of the "pure" organic fraction and SOM is soil organic matter concentration expressed here in g g^{-1}.

Adequacy of the Regression Models

To reduce Type I errors (i.e., falsely rejecting the null hypothesis, and thus incorrectly concluding that a variable should be omitted from the model), data were always partitioned to allow benchmarking. Vanclay and Skovsgaard (1998) and Pothier and Savard (1998) proposed the simple, but efficient, technique of linear regression of observed versus predicted data to evaluate model performance, and to use r^2, the bias and the root mean square error of prediction, to evaluate prediction quality. The accuracy of the equations was evaluated with the statistical terms MBIA (mean bias) and RMSEP (root mean square error of prediction):

$$MBIA(\%) = 100 \times \frac{\frac{\sum_{i=1}^{n}(\hat{y}_i - y_i)}{n}}{\bar{y}} \quad (7)$$

$$RMSEP(\%) = 100 \times \frac{\sqrt{\frac{\sum_{i=1}^{n}(\hat{y}_i - y_i)^2}{n}}}{\bar{y}} \quad (8)$$

where $\hat{y}_i$ is the predicted value, y_i is the observed value, n is the number of observations and $\bar{y}$ is the mean of the observed values.

To test the significance of the bias, a simultaneous F-test for slope = 1 and intercept = 0 was used, as in Yang et al. (2004).

RESULTS AND DISCUSSION

Soil Organic Matter and Organic Carbon Relationship

SOC_{DC} was highly correlated with SOM and could be estimated from the regression

$$SOC_{DC} = 0.4724 \times SOM \; (r^2 = 0.97, \; p < 0.0001) \quad (9)$$

The factor for converting SOM to SOC_{DC} is given by the slope value of Eq. 9, i.e., 0.472 ± 0.007. This value is outside the range of values reported by David (1988; 0.51–0.65), but within the range reported by Broadbent (1953; 0.40–0.58). It is very similar to the range reported by Lowther et al. (1990) for sandy soils in Australia, but is substantially different from the commonly used van Bemmelen factor (0.58). Using a factor of 0.58 overestimates predicted SOC_{DC} by 20% (accuracy = 29%; Table 2), whereas using a value of 0.47 resulted in a 12% underestimation of predicted SOC_{DC} (accuracy = 22%; Table 2). As observed in other studies (Nelson and Sommers 1982; Huntington et al. 1989; Westman et al. 2006), the SOC_{DC}/SOM ratio value was influenced by sampling depth and the best model predicting SOC_{DC} from SOM was the one in which sampling depth was considered (Fig. 1). On average, this model was practically unbiased (slightly underestimated by 3%) with an error of prediction of 16%. As for the model 9, which does not take into account sampling depth, the value of the intercept was not significantly different from zero ($P = 0.067$) so the regression was forced through the origin. The non-significant intercept agreed with certain published results (Spain et al. 1982; Lowther et al. 1990), and disagreed with others (Grewal et al. 1991; Wang et al. 1996; Konen et al. 2002). It indicates that (i) the removal of structural water (hygroscopic and interlattice water) (De Leenher et al. 1957; Goldin 1987; Howard and Howard 1990), (ii) CO_2 release from carbonates (De Leenheer et al. 1957; Goldin 1987; Howard and Howard 1990), (iii) the loss of H_2O from hydroxyl groups in sesquioxides (Goldin 1987) and (iv) destruction of elemental C such as charcoal (Ball 1964) were not important artefacts in these acidic loamy to sandy loam soils. This is consistent with the relatively low clay content of these soil samples. De Vos et al. (2005) found that when the intercept model was significant, it was correlated ($r^2 = 0.86$) with the clay content.

To 50 cm in depth, the SOC_{DC}/SOM values decreased with increasing depth, following a significant ($P = 0.013$) quadratic trend (Fig. 2), from 0.491 at the mineral soil surface to 0.271 at 50 cm. As observed by Federer (1983), the concentration of SOC_{DC} decreased more rapidly with depth than did SOM (Fig. 3). The decline in SOC_{DC}/SOM with depth, also observed by Westman et al. (2006) on pristine forest soils in Finland, suggests a qualitative change in the nature of SOM. Studies of soil organic matter reviewed by Schnitzer and Khan (1978) showed that the chemical composition of SOM changes

Table 2. Some statistics illustrating model prediction errors. A * indicates that $P \leq 0.01$

Models	SOC_{DC} predicted	r^2	MBIA	RMSEP
	(g kg^{-1})		(%)	
Relationships between SOC_{DC} and SOM				
M1: $SOC_{DC} = 0.5800 * SOM$	4.880	0.93	20*	29
M2: $SOC_{DC} = 0.4724 * SOM$	3.560	0.93	−12*	22
M3^z: $SOC_{DC} = \beta_{1depth} * SOM$	3.818	0.95	−3*	16
Relationship between SOC_{DC} and SOC_{WD}				
M4^y: $SOC_{DC} = -0.08208 + \beta_{depth} * SOC_{WD}$	3.915	0.98	0	11

[z] The slope value depends on the depth strata ($\beta_{0\text{–}5} = 0.4908$, $\beta_{5\text{–}10} = 0.4624$, $\beta_{10\text{–}15} = 0.4878$, $\beta_{15\text{–}20} = 0.4437$, $\beta_{20\text{–}25} = 0.4282$, $\beta_{25\text{–}30} = 0.3982$, $\beta_{30\text{–}35} = 0.3844$, $\beta_{35\text{–}40} = 0.3687$, $\beta_{40\text{–}45} = 0.3239$, $\beta_{45\text{–}50} = 0.2712$).

[y] The slope value β_{li}depends on the depth strata i. ($\beta_{1_{0-5}} = 0.9865$; $\beta_{1_{5-10}} = 1.0483$; $\beta_{1_{10-20}} = 1.1236$; $\beta_{1_{20-40}} = 1.1572$; $\beta_{1_{40-60}} = 1.1918$)

with soil depth. In particular, the ratio of oxygen (O) to C increases with depth, indicating that the relative content of O-containing functional groups (C=O, COOH, COH) increases with depth. Schnitzer and Khan (1978) suggested a scheme for the formation of humic substances during SOM degradation that may explain why the O/C ratio increases with depth. Humic acids are higher in molecular weight and lower in O-containing functional groups than are fulvic acids or humin (Tate 1992). The ratio of fulvic acids to humic acids generally increases with soil depth (Kumada 1987).

Relationship Between Soil Organic Carbon Obtained by Wet Digestion and Dry Combustion

The best fit between SOC_{DC} and SOC_{WD} using a linear regression relationship was:

$$SOC_{DC} = -0.08208 + \beta_{1_{depth}} \times SOC_{WD} \tag{10}$$

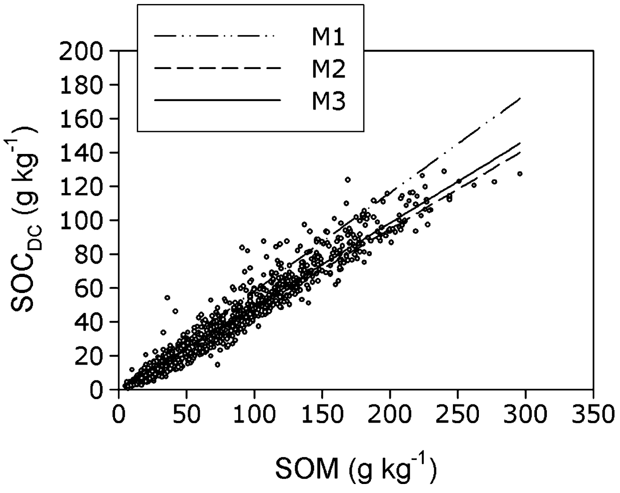

Fig. 1. Soil organic carbon determined by dry combustion as a function of soil organic matter. Equations are presented in Table 3. Dash dot line (M1) uses the van Bemmelen factor, dashed line (M2) represents empirically adjusted, and solid line (M3) represents empirically adjusted taking sampling depth into account.

The negative intercept was the same for all depth strata, and it was significantly different from zero, which is not surprising, as wet digestion is suspected to recover organic C incompletely. Slope parameters, $\beta_{1_{depth}}$, increased following a linear trend ($P < 0.001$) from 0.99 in the upper soil depth strata to 1.19 in the deeper strata (Table 2). The MBIA statistic showed that taking into account both sampling depth and SOC_{WD} allowed SOC_{DC} to be predicted without bias and with an accuracy of SOC_{DC} estimates of 11% (Table 2). A paired t-test indicated that for each soil depth strata there was a significant difference, at a 95% confidence level, between the two methods used to determine SOC. Except for the upper soil depth strata (0–5 cm), the wet digestion method underestimated C concentrations in comparison with the dry combustion method. Additionally, the percentage of recovery decreased with increasing depth (Fig. 4), but it was nevertheless higher than most published values, even in the deeper soil strata where recovery was the lowest (83%). Although soil particle size analyses were not available for the samples used to assess the percentage of recovery of SOC_{WD}, we

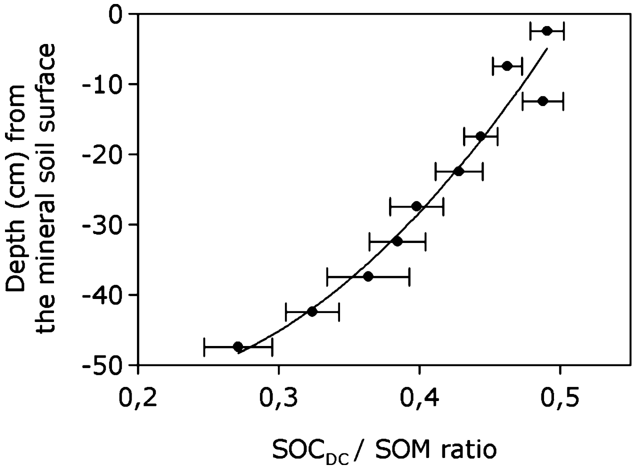

Fig. 2. Mean SOC_{DC}/SOM ratio versus depth in soil profiles, by depth strata of 5 cm. Bars represent standard errors of the means.

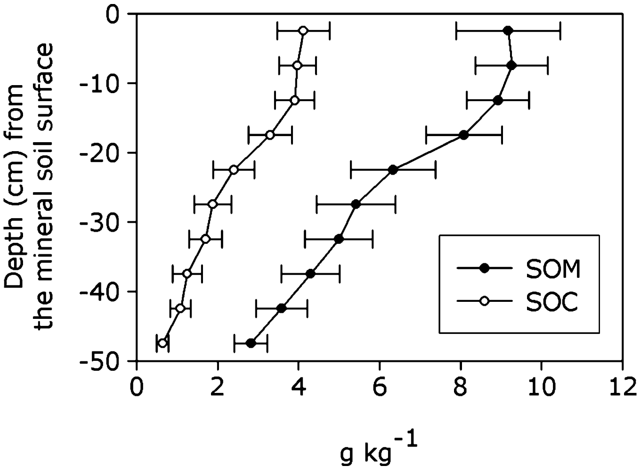

Fig. 3. Soil organic matter (SOM) and soil carbon (SOC) determined by dry combustion plotted versus soil depth. Bars represent standard errors of the means.

did not attribute its reduction with depth to changes in soil texture. For the surficial soil layers, the mean recovery of 98% of total C (SOC_{DC}) by the wet digestion method was high and close to the recovery indicated by Lowther et al. (1990) for podzolic sands or by Jolivet et al. (1998) for loam to loamy sand soils. The reduction in percentage of recovery of SOC_{WD} with depth can be attributed to changes in organic matter composition. Degradation of organic matter in the environment is a continuous process that gradually and selectively modifies the chemical character of soils. As decomposition proceeds, less reactive structures become more dominant in soil organic matter. In Podzols, translocation of organic matter from upper horizons to deeper horizons results in the accumulation of highly decomposed organic matter, which is mainly composed of recalcitrant aliphatic structures. Hatcher et al. (1983) suggested that the increase in the proportion of aliphatic structures as decomposition proceeds is the result of the utilization of easily decomposable carbohydrates by soil microbes, and the selective preservation of more recalcitrant aliphatic structures associated with original plant biopolymers such as cutin, suberin, and waxes. The accumulation of these recalcitrant aliphatic structures with depth (Ussiri and Johnson 2003) could explain the higher extraction efficiencies of wet digestion in the 0- to 20-cm layer than in strata deeper than 20 cm. The high recovery observed in this study was also attributable to the nature of these loamy to sandy loam soil strata, where virtually none of the organic matter present was stabilized as organic mineral complexes in soil aggregates (Lettens et al. 2007). Moreover, the high recovery of C by the wet digestion method (recovery >87%), indicated that this technique could be used in these soils for assessment of *SOC*, especially in the upper 20 cm of the mineral soil.

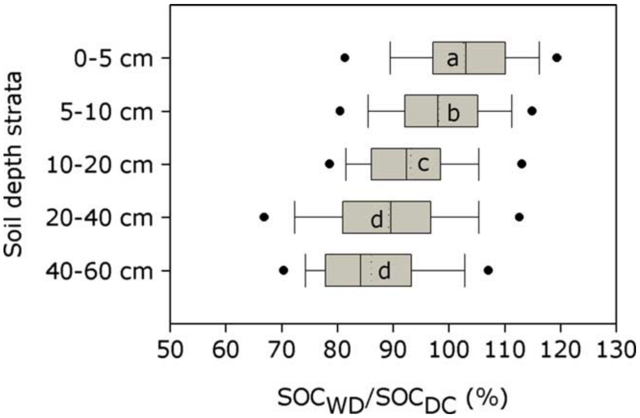

Fig. 4. Effect of depth sampling on the recovery of total carbon by wet digestion method. Box plots with similar letters are not significantly different ($P > 0.05$). Solid line represents median value, dotted line represents mean value and circles represent the 5th and 95th percentiles.

Soil Organic Matter and Bulk Density Relationship

In soil science, two model forms are used to predict mineral soil bulk density from organic matter concentration. One form includes polynomial models like those developed by Curtis and Post (1964), Federer (1983) and Huntington et al. (1989), while the other form, developed more recently by Federer et al. (1993) and used by Post and Kwon (2000), Tremblay et al. (2002) and Prévost (2004), groups nonlinear models based on the organic density concept. In the present study we tested both model forms. Because soil BD and SOM were not estimated on the same basis (soils used to determine BD were air-dried whereas soils used for SOM determination were oven-dried), we examined the relationship between soil moisture content and SOM. SOM and soil moisture content were strongly correlated ($r = 0.98$, $P < 0.001$), so it was possible to correct BD values on an oven-dry basis using SOM to predict soil moisture content. The correction decreased BD values by a maximum of 0.04 g cm^{-3}.

The polynomial equation using SOM, ln(SOM) and $(\ln(SOM))^2$ differed slightly in shape from the equation based on the organic density concept, especially for SOM concentrations greater than 10% (Fig. 5, solid lines). However, accuracies of polynomial and nonlinear models developed in this study were similar (Table 3). The MBIA statistics showed that both equations allowed the soil BD to be predicted without bias. The RMSEP statistic established the mean error of individual soil BD values at 0.149 and 0.154 g cm^{-3}, for the polynomial and nonlinear models, respectively, which correspond to 13.8 and 14.3% of actual values. Considering that direct BD measurements are very laborious for forest soils, these accuracy levels appear to be acceptable for most applications.

The empirically determined D_{bm} and D_{bo} by the nonlinear model were 1.767 ± 0.066 and $0.111 \pm$

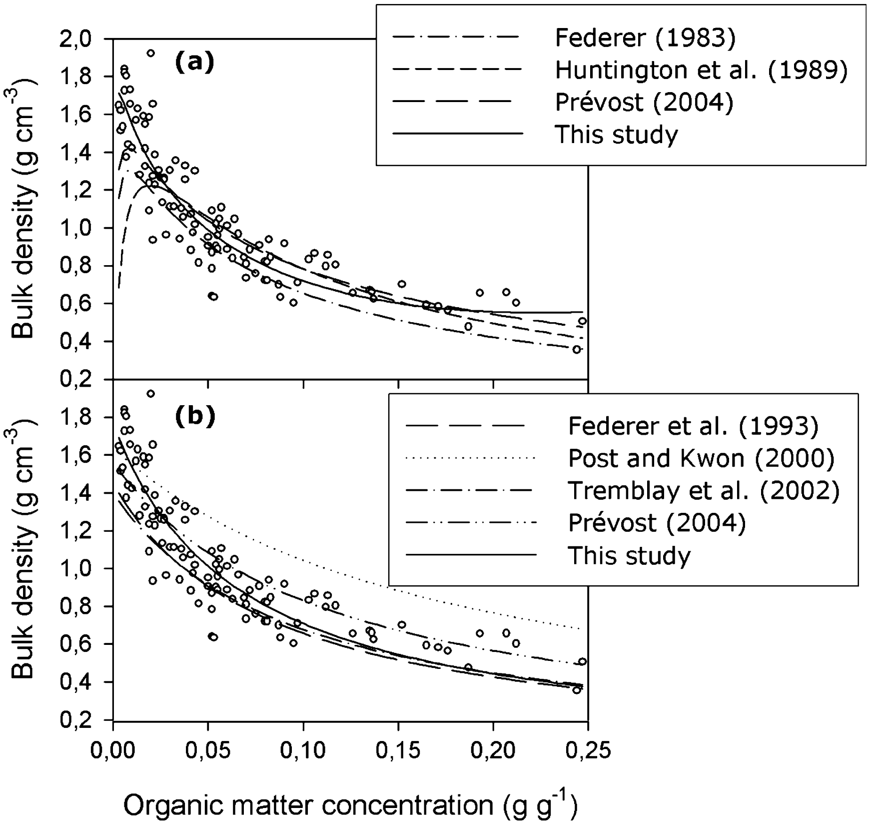

Fig. 5. Scatter plot of soil bulk density (BD) versus soil organic matter (SOM) concentration. The equations obtained from the present study are illustrated by a solid line whereas published equations are illustrated by broken lines, for polynomial models (a) and nonlinear models (b). Regression equations are presented in Table 3.

0.007 g cm^{-3}, respectively. The obtained D_{bm} estimate was higher that reported by Tremblay et al. (2002; 1.40 ± 0.03 g cm^{-3}), but was within the range reported by Federer et al. (1993; 1.45–2.19 g cm^{-3}). The D_{bo} estimate in the present study was exactly the same as that found by Tremblay et al (2002) and Federer et al. (1993) (0.11–0.12 g cm^{-3}).

To verify if it was necessary to adjust the parameters to our data, or if it was possible to use published parameters, we compared BD measurements with predicted BD values obtained by comparing published regression equations for the SOM-BD relationship (Federer 1983; Huntington et al. 1989; Federer et al. 1993; Post and Kwon 2000; Tremblay et al. 2002;

Table 3. Relationships between soil bulk density (BD, g cm^{-3}) and soil organic matter concentration (SOM, g g^{-1}) and some statistics to evaluate the performance of the different methods. The resulting curves are presented in Fig. 5. A* indicates that P ≤0.01.

Reference	Model	$\hat{Y}^z$	r^2	MBIA (%)	RMSEP (%)
Linear Models					
Federer (1983)	$\ln BD = -2.31 - 1.079 \times \ln(SOM) - 0.113 \times [\ln(SOM)]^2$	1.025	0.78	–12*	20
Huntington et al. (1989)	$\ln BD = -2.39 - 1.316 \times \ln(SOM) - 0.167 \times [\ln(SOM)]^2$	1.035	0.42	–6	26
Prevost (2004)	$\ln BD = -1.81 - 0.892 \times \ln(SOM) - 0.092 \times [\ln(SOM)]^2$	1.020	0.78	–1*	16
This study	$BD = -1.977 + 4.105 \times \mathrm{SOM} - 1.229 \times \ln(SOM) - 0.103 \times [\ln(SOM)]^2$	1.063	0.82	0	14
Non Linear Models					
Federer et al. (1993)	$BD = (1.111 \times 1.450)/(1.450 \times \mathrm{SOM}) + (1 - \mathrm{SOM}) \times 0.111$	0.936	0.80	–12*	20
Post and Kwon (2000)	$BD = (1.244 \times 1.640)/(1.640 \times \mathrm{SOM}) + (1 - \mathrm{SOM}) \times 0.244$	1.276	0.75	20*	27
Tremblay et al. (2002)	$BD = (1.120 \times 1.400)/(1.400 \times \mathrm{SOM}) + (1 - \mathrm{SOM}) \times 0.120$	0.937	0.79	–12*	20
Prevost (2004)	$BD = (1.159 \times 1.561)/(1.561 \times \mathrm{SOM}) + (1 - \mathrm{SOM}) \times 0.159$	1.101	0.78	4*	17
This study	$BD = (1.111 \times 1.767)/(1.767 \times \mathrm{SOM}) + (1 - \mathrm{SOM}) \times 0.111$	1.064	0.81	0	14

$^z\hat{Y}$ was backtransformed for logarithmic models.

Prévost 2004). A scatter plot of the data from the present study, superimposed with the published equations, showed a relatively close fit between the equations and the data (Fig. 5). The coefficients of determination varied between 0.42 and 0.82 and eight of the nine models illustrated in Fig. 5 have an $r^2 = 0.75$, which is similar to published results (Huntington 1989, Prévost 2004). However, the seven published models were biased. Five of them underestimated BD values (bias varied from 1 to 12%), whereas two models overestimated BD values (biases were 4 and 20%, respectively; Table 3). Five of the seven published models obtained BD estimates within 20% of the observed values. However, two published models were less adjusted to data from the present study, i.e., the polynomial model of Huntington et al. (1989) and the nonlinear model of Post and Kwon (2000); both were biased and RMSEP was around 30% (Table 3). Of the seven models published, those developed by Prévost (2004) on sandy till soils in the Quebec boreal forest seemed to be the best adjusted to data from the present study with a slight bias (1% for the polynomial model and 4% for the nonlinear model, and prediction errors of 16 and 17%, respectively). Generally, published polynomial models poorly predict BD for soils with organic matter concentrations lower than 1%, which is not the case for the nonlinear models. There is a mathematical reason for this. The published polynomial models are second-order polynomial functions and have a negative coefficient associated with the quadratic parts of the models, indicating an inverse parabola. In this case, when ln(BD) estimates are backtransformed to obtain SOM estimates in the original scale, the BD estimates that are lower than the SOM values observed at the maximum of the parabola will start decreasing with a decrease in SOM. A non-monotone function, like a quadratic function, transformed by a monotone function, like an exponential function, yields a non-monotone function. To preserve the increase of BD with the decrease of SOM, the estimation of ln(BD) should be restricted to the monotone part of the quadratic function, i.e., for SOM values greater than the maximum value of the function.

Implication for Soil Sampling

Sampling soils at 5-cm intervals is a tedious task and many soil scientists prefer sampling soils by genetic horizons, particularly Podzols, which have usually contrasting horizon colors. For the forest soils sampled in this study, most mineral genetic horizons were thicker than the sampling depth interval (5 cm): 6 cm for the A horizon, and 11 cm for the diagnostic B horizon on average (Table 1). However, the relatively constant organic C concentrations observed in the first 10–15 cm of the mineral soil in these profiles may be due to mixing of the thin upper horizons, which may preclude the extremely low organic matter concentrations usually measured in eluviated Ae horizons. In deeper horizons, the relationship between SOC or SOM and soil depth for these soils showed that these variables experienced a fairly continuous and smooth change that increased with soil depth from one 5-cm interval to another (see Figs. 2 and 3). Such a smooth change in such a small depth interval would not have been perceptible if sampling was carried out by genetic horizon. Sampling by genetic horizons, particularly the deeper ones, therefore mixes apparently homogenous, but chemically different soil, in terms of SOC or SOM composition and concentration. This layer mixing may not be a problem if care is taken to sample the whole genetic horizon from bottom to top (to minimize contamination from upper horizons) and not just from the middle of the horizon, in order to cover the within-horizon gradient of SOC and SOM concentrations. For soils similar to the ones sampled in this study, and when soils are sampled by horizons, it is recommended to divide a thick genetic horizon into smaller horizons (for example, a 30-cm Bf horizon divided in two 15-cm Bf1 and Bf2 horizons), in order to characterize some of the hidden variation with depth that was observed by sampling at 5-cm intervals.

These relationships between SOC_{DC} and SOM or SOC_{DC} and SOC_{WD} can be useful for assessing organic C stocks of soils. However, it should be noted that they only apply to the fine earth fraction of soils (<2 mm). Coarse fraction analysis was not included in this paper, although this compartment may represent substantial amounts of organic C (Harrison et al. 2003; Homann et al. 2004).

CONCLUSION

The present study reports relationships between soil organic carbon (SOC), organic matter (SOM), and bulk density (BD), in acidic loamy to sandy loam fine fractions (<2 mm) of forest soils in Quebec (Canada). Our data suggest that in these soils:

i. LOI can be used for determination of total organic C, on the condition that a specific SOC_{DC}/SOM ratio is applied, since SOC_{DC}/SOM value significantly decreased with increasing depth from 0.49 to 0.27, suggesting a qualitative change in the nature of SOM.

ii. The mean bias statistic showed that taking into account both sampling depth and SOC measured by wet digestion allowed total organic carbon to be predicted without bias ($p = 0.548$), and with an accuracy of SOC_{DC} estimates of 11%. The high recovery rate of C by the wet digestion method ($\geq 83\%$), especially in the upper 20 cm of the mineral soil (recovery $>90\%$), indicated that this technique can be used for assessing total organic C in the upper loamy to sandy loam 20 cm soil layer.

iii. BD may be estimated from the SOM concentration measured by LOI, without significant bias and with a degree of accuracy acceptable for most purposes (less than 20%) in these loamy to sandy loam non calcareous forest soils.

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