Soil mineral structural water loss during loss on ignition analyses

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Sun, H., Nelson, M., Chen, F. and Husch, J. 2009. **Soil mineral structural water loss during loss on ignition analyses**. Can. J. Soil Sci. **89**: 603–610. Water loss from soil minerals has been known to cause errors in the determination of soil organic matter when the loss on ignition (LOI) method is used. Unfortunately, no known published studies reliably quantify the range of structural water in the soil. To do this, 15 common reference minerals were analyzed by LOI to obtain their individual water loss. In addition, 14 upland, loamy soil samples and 3 wetland/hydric soil samples with varied mineral contents were analyzed to collect their X-ray powder diffraction spectra. Based upon X-ray spectra peak intensities, the modal abundance of minerals in each soil sample was determined using the RockJock computer program. The resultant modal weight percentages of all identified minerals in each soil sample were then multiplied by the LOI value for each mineral to obtain the mineral structural water loss (SWL) of that soil sample. For the 17 soil samples analyzed, the range of mineral water loss is 0.56 to 2.45%. Depending on the LOI values of the soil samples, the SWL:LOI ratios range from 0.04 to around 1.00. The SWL:LOI ratios are particularly low for top wetland soil when the LOI value is higher. The ratios are lower for surface soil samples than for subsurface soil samples because of the high LOI values in surface soil samples. Understanding soil mineral water loss and its relation to the LOI patterns from various environments is important for the accurate evaluation of soil organic matter when the LOI method is used.

Key words: Mineral, structural water, loss on ignition

Sun, H., Nelson, M., Chen, F. et Husch, J. 2009. **Perte structurelle d'eau par les minéraux du sol lors des analyses de perte par calcination**. Can. J. Soil Sci. **89**: 603–610. On sait que l'eau perdue par les minéraux du sol cause des erreurs lorsqu'on s'efforce de calculer la quantité de matière organique dans le sol par la méthode d'analyse de perte par calcination (LOI). Malheureusement, aucune des études publiées ne quantifie de manière fiable la variation de l'eau structurelle dans le sol. Pour y remédier, les auteurs ont analysé 15 minéraux de référence communs par la LOI afin d'en mesurer la perte d'eau. Ils ont aussi analysé 14 échantillons de sol loameux des plateaux et 3 échantillons de sol hydrique des terres humides afin de recueillir le spectre de leur poudre par diffraction des rayons X. En partant de l'intensité des pics obtenus aux rayons X, ils ont établi l'abondance modale des minéraux dans chaque échantillon grâce au logiciel RockJock. Les pourcentages modaux pondérés des minéraux identifiés dans chaque échantillon qui en résultent ont ensuite été multipliés par la valeur LOI de chaque minéral, ce qui a donné la perte structurelle d'eau (SWL) du minéral pour l'échantillon. Sur les 17 échantillons examinés, la perte d'eau des minéraux varie de 0,56 % à 2,45 %. D'après la valeur LOI des échantillons, le ratio SWL:LOI fluctue de 0,04 à environ 1,00. Le ratio SWL:LOI est particulièrement faible pour le sol de surface des terres humides, où la valeur LOI est plus élevée. Les ratios sont plus faibles pour le sol de surface que pour le sous-sol, en raison de la valeur LOI supérieure des premiers. Il importe de comprendre la perte d'eau des minéraux du sol et leur lien avec la LOI dans divers milieux si l'on veut évaluer correctement la quantité de matière organique dans le sol quand on recourt à la méthode d'analyse de perte par calcination.

Mots clés: Minéral, eau structurelle, perte par calcination

A reliable estimation of soil organic matter (SOM) or soil organic carbon is required for studying agricultural soil reactions and environmental soil water pollution, as soil organic carbon is often the main absorption media of ions and compounds in the topsoil (Drever 1997; Smith 2003; Brady and Weil 2007). In addition, the accurate estimation of SOM in soils is necessary for evaluating the carbon trapping ability of various soil environments and the feasibility of sequestering carbon dioxide as a method of mitigating future global warming (Frogbrook and Oliver 2001; Lal 2004; Fang et al. 2005; Davidson and Janssens 2006).

The loss on ignition (LOI) method has been widely used for estimating soil organic matter, and it typically involves placing a weighed sample in a muffle furnace at 450–550°C for 2.5 to 4 h and then determining the sample's weight loss immediately upon removal (Frogbrook and Oliver 2001; Smith 2003; Vos et al. 2005; Abella and Zimmer 2007). Relative error ranges

Abbreviations: LOI, loss on ignition; **SOM**, soil organic matter; **SWL**, structural water loss; **XRD**, X-ray powder diffraction

for estimation of SOM based on the LOI method have been reported as 2 to 5% and, traditionally, the method's ability to determine soil SOM has been considered reliable (Dean 1974; Howard and Howard 1990; Snowball and Sandgren 1996; Abella and Zimmer 2007). While the LOI method may be appropriate for soil samples with large SOM, as often is found in wetland soils, studies (Konen et al. 2002; Smith 2003) have indicated that soil mineral water loss, particularly the clay minerals, can cause significant errors for upland soils where SOM usually is less than 6%. Unfortunately, there are no known published studies that can reliably quantify this error. Therefore, the purpose of this study is to quantify the structural water loss (SWL) from a soil sample during the LOI process and to facilitate further in-depth discussion on the subject.

STRUCTURAL WATER OF MINERALS
While the structurally bound mineral water can be separated into crystal lattice water for OH– ion and water of hydration for bonded H_2O (Tan et al. 1986), the broad term "structural water" has commonly been used to include both types of water by researchers due to the difficulty of their separation in LOI processes (Howard and Howard 1990; Vos et al. 2005; Schulte 1991; Heiri et al. 2001; Konen et al 2002; Smith 2003). Therefore, following the common practice, the term "structural water" is adopted in this study, which will include all bonded H_2O molecules and OH units in a mineral that can be lost during a heating process. This includes both interlayer water molecules held in place by polar forces between the tetrahedral and octahedral layers and OH units bonded within the brucite- or gibbsite-like octahedral sheets of the clay minerals (Klein and Dutrow 2007). It also includes the water molecules and OH units bonded within the structures of other minerals. The theoretical weight percentages of structural water of 15 reference soil minerals, which are identified in our soil samples in a later section, were calculated based upon their accepted chemical formulae (Klein and Dutrow 2008) and are given in Table 1 as a reference. These data are consistent with the data given by Tan et al. (1986).

Because only part of the theoretical "structural water" can be lost for most minerals during the LOI analysis at $550°C$, the LOI values for each reference mineral will need to be collected experimentally in order to evaluate the SWL of collected soil samples at $550°C$.

METHODS
Loss on Ignition Measurement of Reference Minerals
Fifteen naturally formed reference mineral samples were obtained from a commercial supplier (Wards Natural Science) and used to measure their LOI values at $550°C$. For the non-clay minerals, quartz, feldspar, gypsum, calcite, biotite, muscovite, hematite, goethite, and dolo-

Table 1. Weight percent of theoretical "structural water" of reference soil minerals calculated based upon their chemical formulae (Klein and Dutrow 2008)

Mineral names	Chemical formula	% of theoretical H_2O and OH
Chlorite	$(Mg,Fe)_3(Si,Al)_4O_{10}(OH)_2 \cdot (Mg,Fe)_3(OH)_6$	20.35
Illite	$(K,H_3O)(Al,Mg,Fe)_2(Si,Al)_4O_{10}[(OH)_2,(H_2O)]$	12.03
Kaolinite	$Al_2Si_2O_5(OH)_4$	13.96
Smectite	$(Na,Ca)Al_4(Si,Al)_8O_{20}(OH)_4 \cdot 2(H_2O)$	9.19
Vermiculite	$(Mg,Fe^{++},Al)_3(Al,Si)_4O_{10}(OH)_2 \cdot 4(H_2O)$	17.87
Biotite	$K(Mg,Fe++)_3AlSi_3O_{10}(F,OH)_2$	3.64
Muscovite	$KAl_2(Si_3Al)O_{10}(OH,F)_2$	4.07
Gypsum	$CaSO_4 \cdot 2(H_2O)$	20.93
Goethite	$FeO(OH)$	10.14
Calcite	$CaCO_3$	0.0
Dolomite	$CaMg(CO_3)_2$	0.0
Hematite	Fe_2O_3	0.0
K-Feldspar	$KAlSi_3O_8$	0.0
Na,Ca-Feldspar	$(Na, Ca)(Si,Al)_4O_8$	0.0
Quartz	SiO_2	0.0

mite samples were selected from relatively well-formed crystals before being crushed for the LOI procedure to limit their impurities to an insignificant level.

For the reference clay minerals kaolinite, vermiculite, smectite, illite, and chlorite, because mixtures of other minerals commonly exist in nature, quantitative X-ray powder diffraction (XRD) analyses (see procedures in a later section) were conducted to obtain the modal abundance (weight percentage) of the minerals. The LOI of individual clay mineral was then corrected based on their mineral modal abundance and the sample LOI value.

All 15 minerals with dual samples were heated in a muffle furnace at $550°C$ for 4 h after being dried at $105°C$ overnight.

Characters of Soil Samples and Measurement of their Loss on Ignition
Fourteen upland soil samples and three wetland/hydric soil samples from New Jersey, on the east coast of the United States, were collected using soil hand augers. Samples with the prefix DA (Ultisols) and DB (Alfisols) were collected from areas where a slight surficial soil disturbance exists; samples with the prefix MCP (Alfisols) were collected from wooded areas where no soil disturbance exists; samples with the prefix I (Inceptisols) were collected from a site where serious soil disturbance exists; and samples with the prefix MA (Histosols) were collected from a brackish marsh. The sites of upland samples are underlay by non-calcareous clastic sedimentary rocks and the site of wetland samples is underlay by a sand layer. The selection of the soil samples at varied depths of upland and wetland was to reflect the various amounts of SOM and clay minerals under different environmental conditions.

For this study, samples collected at a depth less than 20 cm are roughly classified as surface soil sample and samples collected at a depth greater than 20 cm are roughly classified as subsurface soil sample. All upland samples were collected from the A and B soil horizons at a depth of less than 108 cm. Loss on ignition of soil sample was measured in the same manner as for the 15 references minerals. The air-dried soil samples were dried in a vacuum oven for at least 24 h before being placed in a muffle furnace.

The Mineral Modal Abundance and Structural Water Loss of Soil Samples

In order to calculate the SWL of soil samples based upon their mineral modal abundances (i.e., the weight percentage), these latter values had to be determined utilizing a quantitative X-ray powder diffraction method. The SWL, which is categorized as the weight loss resulting from all non-organic materials in the soil sample during LOI at 550°C, will be calculated from the summed products of the mode and the LOI at 550°C for each mineral.

Quantitative XRD for Determining Mineral Modes

Quantitative XRD analysis for determining mineral modes in rock and soil samples is a common practice (Maniar and Cooke 1987; Moore and Reynolds 1997; Ward et al. 1999). A quantitative EXCEL macro program, RockJock, developed by Eberl (2003) was selected to do this. The program uses the measured peak intensities as input data and calculates the modal abundance (in weight percent) of all identified minerals using zincite as an internal standard. The theory behind the RockJock program was first described by Srodon et al. (2001), with the program being made public by Eberl (2003). The program placed third in the 2002 Reynolds Cup competition, sponsored by The Clay Minerals Society, for the most accurate quantitative phase analysis (McCarty 2002); it has been improved significantly since that time (Eberl 2004). Typical program results are within 1 to 2 weight percent of actual mineral abundance values (Eberl 2003, 2004). The program was recommended by Mertens et al. (2006) as "the method of choice" for the quantification of clay minerals in soil

samples after it was compared was the Rietveld approach (Topas Academic by Coelho Software). For this study, we utilized the RockJock6 version, dated 6/10/2008.

Following the sample preparation procedures described by Srodon et al. (2001) and Eberl (2003), 2 g of each sample was mixed with 0.222 g of zincite (ZnO) and placed in a SPEX mixer/ball mill with 5 mL of methanol added. The mixture was shaken for 10–15 min before being removed and dried in a vacuum oven. The dried samples hardened into a durable crust; therefore, samples were first disaggregated and mixed with a spatula, then sieved through a 180 μm mesh (Standard Sieve No. 80), and finally adhered onto glass slides with petroleum jelly. X-ray diffraction data for the samples were collected from 5 to 65° two-theta using Cu K-alpha radiation and a step-size of 0.02°.

Structural Water Loss of Soil Samples

Once the modes of the major minerals are measured, the SWL of minerals in a soil sample can be calculated as the summation of the modal abundances of minerals multiplied by the SWL of each individual mineral:

$$SWL = \sum_n SWL_{Mineral} \times Weight\ Percent_{Mineral} \qquad (1)$$

$Weight\ Percent_{mineral}$ is the weight percent of a mineral measured in a soil sample by the RockJock program; $SWL_{mineral}$ is the measured LOI of that mineral; and n is the total number of minerals measured in a soil sample.

RESULTS

Loss on Ignition Results of Fifteen Reference Minerals

Loss on ignition results for the 15 reference minerals utilizing discussed heating procedures are reported in Table 2. For the LOI of each clay mineral, a correction was made based upon the modal abundances of minerals (Table 3) and the measured LOI for each clay sample by solving the multiple equations aided by MATHEMATICA (Wolfram Software).

Table 2. Measured weight loss (LOI%) at 550°C in a muffle furnace after the samples were dried at 105°C overnight

Mineral name	Weight loss at 550°C (%)	Mineral name	Weight loss at 550°C (%)
Chlorite	1.23[z]	Goethite	2.45
Illite	1.72[z]	Dolomite	2.02
Kaolinite	18.4[z]	Calcite	0.13
Smectite	0.95[z]	Hematite	0.17
Vermiculite	12.6[z]	K-Feldspar	0.14
Biotite	0.46	Na,Ca-Feldspar	0.08
Muscovite	0.51	Quartz	0.12
Gypsum	15.84		

[z]Indicates the corrected values of LOI for clay minerals. The initial LOI values of the samples before the correction for impurities are illite 2.08, kaolinite 12.47, smectite 1.69, vermiculite 5.75, chlorite 1.209.

Table 3. Mineral modes (%) of clay samples

Chlorite sample		Illite sample		Kaolinite sample	
Non-clay		Non-clay		Non-clay	
		Quartz	17.39	Feldspar	7.00
Total non-clays	1.50	Total non-clays	19.70	Total non-clays	9.68
Clays		Clays		Clays	
Chlorite	97.01	Kaolinite	5.69	Kaolinite	66.61
		Illite	45.97		
		Smectite	23.66	Smectite	22.22
Total clays	98.55	Total clays	80.26	Total clays	90.32
Smectite sample					
		Vermiculite sample			
Non-clay		Non-clay			
Quartz	5.56	Feldspar	10.06		
Feldspar	8.17				
Total non-clays	16.17	Total non-clays	13.74		
Clays		Clays			
Kaolinite	5.16	Vermiculite	41.08		
Smectite	77.67	Chlorite	42.67		
Total clays	83.83	Total clays	86.26		

Note. Minerals with weight percent less 4% are not listed.

The LOI values for clay minerals are consistent with the data patterns reported in previous thermal analysis studies of minerals (Barshad 1965; Tan et al. 1986). In particular, there is a relatively large percentage of water loss by kaolinite during the LOI at 550°C, which is associated with its phase transformations upon thermal treatment. This water loss was reported as the dehydration (or endothermic dehydroxylation) of kaolinite that begins at 500–600°C to produce disordered metakaolinite, $Al_2Si_2O_7$ (Tan et al. 1986; Bellotto et al. 1995). A moderately large amount of water loss at 550°C by vermiculite is probably due to both the endothermic dehydroxylation (peaked at 500°C) and loss of absorbed water as reported by Tan et al. (1986). The relative small amounts of LOI at 550°C for the other three clay minerals (illite, chlorite and montmorillonite) are probably due to the fact that there is only the loss of adsorbed water and no significant dehydration loss of OH under 550°C for these three minerals (Barshad 1965; Tan et al. 1986). Because varied cation saturation levels can affect the amount of layer water, a deviation of LOI may exist for a natural clay mineral (Barshad 1965; Tan et al. 1986; Moore and Reynolds 1997). For the non-clay minerals, gypsum, goethite, and dolomite are the minerals measured that have relatively high LOI values. Santisteban et al. (2004) suggested LOI below 550°C for dolomite was due to the breaking off of inorganic carbon from the dolomite crystalline structure as CO_2. In addition, a small amount of weight loss, <0.2%, was recorded during LOI analysis at 550°C for quartz and feldspar. This small LOI for quartz and feldspar is consistent with the data reported by Tazaki et al. (1992), where LOI for feldspar was <0.4% and <0.1% for quartz. A small LOI weight loss of 0.134% at 550°C was also recorded for calcite, indicating that there is little, if any, inorganic carbon breaking off the calcite structure. This is similar to the findings of Dean (1974) and Heiri et al. (2001) of "no significant LOI for calcite."

Loss on Ignition Result of Soil Samples
Utilizing the same heating procedure as for the reference minerals, LOI values for collected soil samples are given in Table 5 (together with SWL and SWL:LOI ratio). For samples with apparent larger amount of SOM, there is a larger LOI, such as MA0-19 and MA80-100, which is similar to the previous studies (Konen et al. 2002; Jankauskas et al. 2006; Abella and Zimmer 2007). The overall LOI values for surface soil samples are larger than those for subsurface soil samples.

Results for Soil Mineral Modal Abundances and their Structural Water Loss
Overall, the RockJock program's calculated spectra produced a fairly good match to the measured XRD spectra for all the samples (Fig. 1). The modal abundance of soil minerals shows that the most abundant non-clay mineral group in these soil samples is the framework silicate, which includes quartz and feldspar (Table 4). These two species regularly constitute more than 50% of the total mineral content in the 17 soil samples. Other less abundant, non-clay mineral species include iron oxides and hydroxides, carbonates, and salts. Clay minerals constitute approximately 17 to 54% of the total minerals in the 17 soil samples. Identified clay minerals include the tetrahedral-octahedral (T-O) type, kaolinite, and the tetrahedral-octahedral-tetrahedral (T-O-T) types, smectite, illite, chlorite, and mica group. In general, more K-feldspar than Na, Ca-feldspar is found in the soil samples. There are also no statistically significant linear relationships between mineral abundances and sample depth. However, the
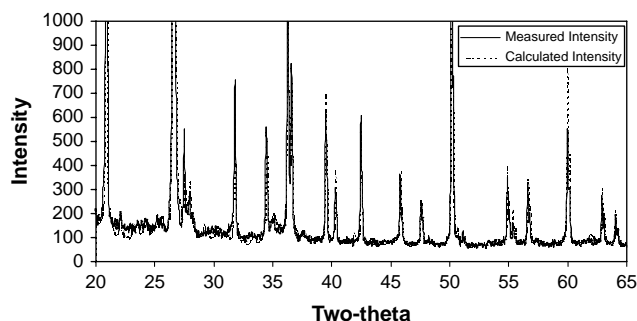
Fig. 1. Full pattern fit of calculated spectrum from RockJock vs. measured XRD spectrum of sample DB72-82.

Fig. 2. SWL:LOI ratio vs. LOI plot and the power fitting equations of the two sets of ratio data.

data indicate clay mineral abundances generally are greater in subsurface soil horizons (depth > 20 cm) than in surface soil horizons (depth < 20 cm).

The LOI, SWL, and SWL:LOI ratios for each soil sample at $550°C$ are given in Table 5. The SWL of the 17 soil samples collected in this study range from 0.56 to 2.45%. The SWL:LOI ratios range from 0.04 to 1.12, depending on the amount of LOI values of the soil samples. For the two top wetland hydric soil samples, which have large number of LOI, the SWL:LOI ratios are less than 0.1. Also, the SWL:LOI ratios for surface soil samples (< 20 cm depth) are smaller than those for subsurface soil samples (20 to 108 cm depth).

DISCUSSION

From the results given in the previous section for mineral LOI (Table 2) and calculated SWL (Table 5), it is clear that soil SWL represents only a small percentage of the soil mass, varying from 0.56 to 2.45% in all our samples. In addition, soil sample SWL depends only on the types and modal abundances of the minerals each sample contains. Therefore, the calculated SWL:LOI ratios will largely depend on the LOI value for a given soil sample (Table 5).

Based upon the LOI data from Table 2, efforts were made to use the quantitative XRD mineral modal abundance reported by Ruffell and Wiltshire (2004) to calculate the SWL and SWL:LOI values for their soil samples. For the 21 surface soil samples collected from various locations by Ruffell and Wiltshire (2004), their SWL ranged from 0.03 to 5.55%. This range of SWL value is comparable to the SWL values from samples in this present study. However, Ruffell and Wiltshire's (2004) samples have a higher average LOI value of 9.8% than the average LOI of 6.67% for samples measured in this study, although Ruffell and Wiltshire (2004) used slightly different temperature steps and heating durations.

The SWL:LOI versus LOI plot (Fig. 2) clearly shows that the SWL:LOI ratio is mainly a function of LOI, as indicated by the best-fit power trend line and the R^2 value of the power equation. The larger the LOI value (likely, a larger SOM), and the less need for SWL
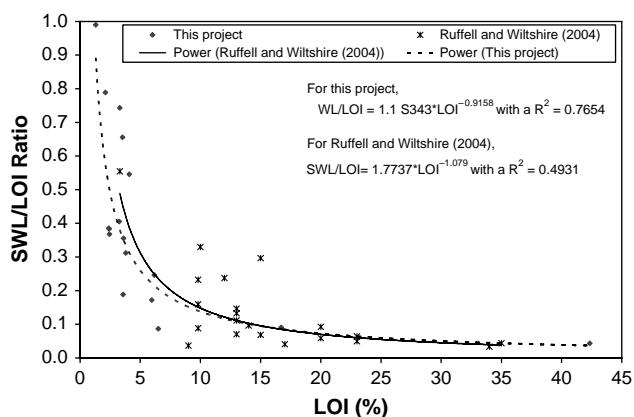
correction in the estimation of SOM. Overall, the general characteristics of clay minerals, the distribution of SOM with the depth, and SWL:LOI ratios in different soil horizons lead to the conclusion that correcting for SWL when estimating SOM by the LOI method is more important for subsurface soil samples than for the surface soil samples. From Fig. 2, one can also deduce that the potential errors of estimating SOM by LOI method can be significantly larger than the 2 to 5% commonly believed error range (Dean 1974; Howard and Howard 1990; Snowball and Sandgren 1996; Abella and Zimmer 2007). For example, if the LOI measurement of a soil sample is 2%, the structural water loss of a soil sample is 2% and one assumes the LOI being purely due to SOM, then the error will be 100%.

The results of this study can also be used to partially explain the differences found in the relationship between LOI and organic carbon or SOM when utilizing LECO and other methods. For example, in the study of Konen et al. (2002), most of the data exhibited good linear relationship between LOI and LECO organic carbon with the coefficients of determination R^2 between 0.94 and 0.98 (their Fig. 2). In the study of Goldin (1987), R^2 for this relationship of mineral soils is 0.86 (Goldin's Fig. 1). In the study of Schulte et al. (1991), using the Walkley-Black method, the R^2 for the relationship between LOI and SOM is 0.90 from their Fig. 1. Generally, a smaller R^2 indicates a larger envelop range along the best fit line and a large potential error for estimating the organic carbon or SOM. From Fig. 1 of Goldin (1987), if LOI is given as 90 g kg^{-1}, the range of LECO carbon can vary from ~ 22 to 39 g kg^{-1} and the difference is $(39-22)/39 = 43.6\%$. From Fig. 1 of Schulte et al. (1991), if LOI is given as 6%, the determined organic matter can range from ~ 4 to 8.7%. The potential error for estimating the SOM can be large.

The soil samples for the study of Konen et al. (2002) were from north-central United States. This region is generally dry, and there are limited hydrous oxides and relatively abundant mixed and smectite clays (Brady and

Table 4. Mineral modes (%) of soil samples

	Quartz	K-feldspar	Na, Ca-feldspar	Calcite	Dolomite	Gypsum	Hematite	Goethite	Kaolinite	Smectite	Illite	Biotite	Chlorite	Muscovite
DA0-19	70.1	4.45	2.94	0.3	0	0.97	0.45	0.7	4.04	5.01	2.68	1.09	3.43	1.02
DA41-47	68.66	7.47	0	0	0	0	0.16	1.56	5.49	7.47	1.97	0.66	2.82	1.5
DA61-67	49.1	5.64	0	0.01	0.64	0	0	1.68	9.86	15.24	6.6	1.58	4.59	3.26
DA91-105	53.1	5.34	4.32	0	0	0.53	0	6.03	2.09	9.02	8.29	2.25	2.98	0.95
DB0-19	63.6	3	4.64	0	0.4	0	0.2	1.51	1.03	6.86	3.93	1.88	4.47	2.38
DB30-45	59.06	3.47	3.02	0	0.14	0	0.56	1.65	2.5	10.82	5.4	2.61	6.63	2.12
DB72-82	62.92	4.23	4.49	0	0	0.8	0	0.85	2.42	12.44	2.89	3.01	3.79	0.6
DB105-108	59.88	5.64	2.93	0	0.15	0.05	0.72	0.71	2.79	10	6.25	3.36	4.2	1.5
I8-19.5	56.45	3.22	0.84	0	0.36	1.31	0.06	1.23	5.67	10.53	3.41	4.65	7.05	3.89
I27-32	59.07	3.74	0	0	0.01	0	0.01	2.27	9.6	13.19	7.42	0.56	1.75	0
I32-36	39.71	5.67	0	0	0.49	0.57	0.4	2.77	9.83	20.46	2.75	2.85	8.83	4.11
MCP0-15	58.87	5.4	11.31	0	0.48	0.35	0.2	0.12	1.74	12.93	0.71	0.74	2.56	1.68
MCP30-45	42.63	3.61	13.25	0	0.13	0	0.6	1.76	3.84	17.62	5.18	1.01	4.52	2.88
MCP40-50	37.52	9.33	10.1	0	0.12	0.38	0	2.92	5.39	18.19	0.06	2.61	3.55	4.79
MA0-19	6.01	13.48	3.46	0	0.17	0.82	0	0.93	3.24	13.72	4.02	3.4	4.75	4.44
MA80-100	10.87	19.36	4.84	0	0	1.59	0.57	1.21	3	21.59	3.43	5.76	7.84	3.8
MA300	12.73	22.63	5.61	0	0	1.9	0.64	1.57	3.23	23.78	2.86	7.57	10.17	6.41

Note: DA, Drexel Woods site A; DB, Drexel Woods site B; I, Near highway I-95 disturbed soil site; MCP, Mercer County Park undisturbed soil site. MA, Marsh soil site in southern NJ. Sample numbers listed after site identification letters give the sample depth interval in centimeters.

Table 5. LOI (%) at 550 °C, SWL (%), and SWL/LOI ratios of 17 soil samples

	SWL(%)	LOI(%)	SWL:LOI
DA0-19	1.28	3.61	0.36
DA41-47	1.32	3.26	0.41
DA61-67	2.31	3.53	0.66
DA91-105	1.02	5.95	0.17
DB0-19	0.56	6.49	0.09
DB30-45	0.90	2.44	0.37
DB72-82	0.92	2.41	0.38
DB105-108	0.92	2.38	0.39
I8-19.5	1.66	2.11	0.79
I27-32	2.23	4.08	0.55
I32-36	2.45	3.30	0.74
MCP0-15	0.67	3.57	0.19
MCP30-45	1.18	3.80	0.31
MCP40-50	1.52	6.17	0.25
MA0-19	1.83	42.34	0.04
MA80-100	1.52	16.71	0.09
MA300	1.45	1.30	1.12

Note: LOI, loss on ignition (%); SWL, structural water loss (%); SWL:LOI, ratio of SWL to LOI. Sample numbers listed after site identification letters are the sample depth interval in centimeters.

Weil 2007). The combination of minerals and high SOM in their soil samples will mean a larger R^2 between the relationship of LOI and organic carbon, and less need for SWL correction during the LOI process. On the other hand, if the soil samples are obtained from a humid climate such as Southern United States and South America or coastal Pacific Canada, as seen in Goldin's (1987) study, there may be more goethite (or other hydrous oxides) and/or kaolinite. The relationship between organic carbon and LOI will exhibit a smaller R^2 and the need for SWL correction will be relatively greater.

SUMMARY AND CONCLUSIONS

The LOI values for 15 common reference soil minerals were determined at 550°C. The LOI for clay minerals, due mainly to their abundant structural water, is more significant than that for the non-clay minerals. Seventeen soil samples collected from different environment settings were used to evaluate SWL during LOI analysis. Soil sample mineral modes were obtained from X-ray powder diffraction and the RockJock program. The summed products of the modal abundance for each soil mineral and corresponding LOI value were computed to obtain the total SWL of a soil sample. The calculated SWL ranged from 0.56 to 2.45% for our soil samples. The SWL:LOI ratios largely varied with the LOI values of the samples, ranging from 0.04 to about 1. The ratio was relatively smaller for wetland soil and surface soil, most likely due to the large amount of the SOM in these environments. The result also indicates that SWL correction in the estimation of SOM is needed more in regions where significant amounts of goethite (a hydrous oxide) and kaolinite and vermiculate (clay minerals) are present in soil.

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