Evaluating Loss-on-Ignition Method for Determinations of Soil Organic and Inorganic Carbon in Arid Soils of Northwestern China[*1]

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ABSTRACT

There is a need for determinations of soil organic carbon (SOC) and inorganic carbon (SIC) due to increasing interest in soil carbon sequestration. Two sets of soil samples were collected separately from the Yanqi Basin of northwest China to evaluate loss-on-ignition (LOI) method for estimating SOC and SIC in arid soils through determining SOC using an element analyzer, a modified Walkley-Black method and a LOI method with combustion at 375 °C for 17 h and determining SIC using a pressure calcimeter method and a LOI procedure estimated by a weight loss between 375 to 800 °C. Our results indicated that the Walkley-Black method provided 99% recovery of SOC for the arid soils tested. There were strong linear relationships ($r > 0.93$, $P < 0.001$) for both SOC and SIC between the traditional method and the LOI technique. One set of soil samples was used to develop relationships between LOI and SOC (by the Walkley-Black method), and between LOI and SIC (by the pressure calcimeter method), and the other set of soil samples was used to evaluate the derived equations by comparing predicted SOC and SIC with measured values. The mean absolute errors were small for both SOC (1.7 g C kg^{-1}) and SIC (1.22 g C kg^{-1}), demonstrating that the LOI method was reliable and could provide accurate estimates of SOC and SIC for arid soils.

Key Words: calcareous soil, dry combustion, linear regression, pressure calcimeter method, Walkley-Black method

INTRODUCTION

Soil organic matter (SOM) plays an important role in the terrestrial ecosystems, not only as a source of nutrients for cropland but also as a means for carbon sequestration (Konen *et al.*, 2002; Rasmussen, 2006). Thus, evaluating SOM and/or soil organic carbon (SOC) have been an important topic for both agricultural science and environmental science. Soil inorganic carbon (SIC), exceeding SOC by a factor of 1–9 on arid and semi-arid lands (Scharpenseel *et al.*, 2000), is starting to attract attention (Denef *et al.*, 2008; Rantakari *et al.*, 2010) because of its potential for carbon sequestration (Lal, 2004). With the increasing interest in soil carbon sequestration for mitigation of the greenhouse effects, there is an undeniable need to find simple and cheap methods for determining SOC and SIC.

Dry combustion (*i.e.*, automated elemental analyzers) and wet chemical oxidation (Walkley and Black, 1934) are the common methods to determine SOM or SOC. Although the automated technique is an accurate and precise method, it is not a common practice in many research laboratories in China because of the high costs. In addition, this approach often involves pretreatment with acid to remove carbonate for calcareous soils, which may destroy organic matter in soil samples and erode the instruments if it is not done properly (Byers *et al.*, 1978). The wet chemical oxidation, *i.e.*, the Walkley-Black method, has been used widely to determination SOC in soils (Nelson *et al.*, 1996), particularly in China (Xu *et al.*, 2007; Gong *et al.*, 2009; Wang *et al.*, 2010; Huang *et al.*, 2012). This technique is less expensive, but more time-consuming than dry combustion. Moreover, this procedure creates hazard Cr as a consequence of dichromate with concentrated sulfuric acid (De Vos *et al.*, 2007). Another disadvantage is that the Walkley-Black method requires a correction factor that may introduce uncertainties for estimates of SOC.

Soil inorganic C content is usually determined by measuring either H^+ or CO_2 production following HCl acid dissolution (Dreimanis, 1962; Presley, 1975; Nômmik and Larsson, 1986). Because some soil components can consume H^+, the method that involves acid addition and back titration is not suitable for some soils, e.g., alkaline soils. Hence, SIC is often determined by measuring CO_2 production under the condition of no SOM decomposition. For instance, Sherrod et al. (2002) reported a modified pressure calcimeter method, with addition of $FeCl_2$ to minimize the production of CO_2 from organic matter.

Alternatively, loss-on-ignition (LOI) that measures weight losses during combustion can provide estimates of SOM or SOC and carbonate (Wang et al., 2011). Relatively, LOI technique is simple, less expensive and less labor intensive relative to the automated techniques and chemical methods. Thus, LOI technique has been used to estimate SOM/SOC in agricultural, forest soils and sediments (Ball, 1964; David, 1988; Konen et al., 2002; De Vos et al., 2005; Wright et al., 2008) and carbonate in sediments (Dean, 1974; Heiri et al., 2001; Santisteban et al., 2004; Wang et al., 2011). Some other studies demonstrated that LOI method can be accurate and precise if it is carried through cautiously (Heiri et al., 2001).

While LOI technique has been widely utilized for determining SOC, it is rarely used for soils in the arid and semi-arid regions. On the other hand, there is little use of LOI technique for SIC estimates. In the arid region, SIC content is 2–10 times as high as SOC (Scharpenseel et al., 2000). Thus, one would think that LOI method may have potential for estimating SIC. The objective of this study was to evaluate LOI method for the determinations of SOC and SIC in the arid land of northwestern China.

MATERIALS AND METHODS

Site description

We conducted a survey at the Yanqi Basin ($42°10'$–$41°55'$ N, $86°20'$–$86°33'$ E) that is located on the southeastern flank of the Tianshan Mountains. According to the Yanqi Meteorological Station's data, mean precipitation is about 75 mm year^{-1}, and evaporation about 2 000 mm year^{-1}. Annual mean temperature is 8.2–9.3 °C, with an annual maximum of 38.8 °C and minimum of -35.2 °C. We collected soil samples from both sides of the Kaidu River, with various land uses, including shrub land, cropland and grassland. Brown desert soil is the main soil type, which was developed from limestone parent material and classified as a Haplic Calcisol (FAO, 1988).

Soil sampling and analyses

In the present study, two sets of soil samples were collected: 70 samples from 14 profiles in August, 2010 (referred to as set 1), and 105 samples from 17 profiles in November, 2010 (referred to as set 2). The latter was used for developing regressions between LOI method and traditional methods (i.e., the Walkley-Black method for SOC, and a pressure calcimeter method for SIC), and the former for evaluating the regressions developed. Soil samples were taken from depths of 0–5, 5–15, 15–30, 30–50 and 50–100 cm. Soils were air-dried, thoroughly mixed, and sieved to pass a 2-mm screen to determine soil pH and electric conductivity (EC) in a 1:5 soil:water extract. Representative subsamples were ground (< 0.25 mm) prior to analyses of SOC and SIC. As shown in Table I, soils from both

TABLE I

Ranges of soil pH, electric conductivity (EC), soil organic carbon (SOC) and soil inorganic carbon (SIC) at various depths, and the contents of clay, silt and sand in 0–30 cm

Soil	Depth	pH	EC	SOC[a]	SIC[b]	Clay	Silt	Sand
	cm		mS cm^{-1}	g C kg^{-1}		%		
Set 1	0–5	7.7–9.1	0.1–36	6–28	15–47	4–9	56–77	16–40
	5–15	7.8–9.8	0.1–9.7	4–23	17–47			
	15–30	7.6–9.8	0.1–10	3–16	20–40			
	30–50	7.6–9.8	0.1–8.6	3–11	23–40			
	50–100	7.7–9.3	0.2–6.2	1–10	23–43			
Set 2	0–5	8.1–9.9	0.1–8.3	4–18	10–48	4–11	69–78	12–25
	5–15	8.2–9.3	0.1–4.9	1–17	9–49			
	15–30	8.1–9.3	0.1–4.3	1–14	9–47			
	30–50	8.0–9.4	0.1–4.3	1–11	8–46			
	50–100	8.7–9.4	0.0–6.2	1–6	7–51			

[a]Determined by the Walkley-Black method.
[b]Determined by the pressure calcimeter method.

sets have a similar range of pH, SOC, SIC and soil texture.

A CNHS-O elemental analyzer (Model EuroEA-3000, EuroVector, Italy) was used for SOC estimates, and 67 soil samples were randomly selected from set 2. A soil sample (20 mg) was pretreated with 10 drips of H_3PO_4 for 12 h to remove carbonate and then combusted at 1 020 °C with constant helium flow carrying pure oxygen to ensure complete oxidation of organic materials. The production of CO_2 was determined using a thermal conductivity detector.

We also used a modified Walkley-Black method for SOC measurement. The traditional Walkley-Black method involves heating soil-H_2SO_4-$K_2Cr_2O_7$ mixtures at 120 °C for 30 minutes (Walkley and Black, 1934). In this study, a soil sample (about 0.4 g) was first treated with 5 mL concentrated H_2SO_4 for 4 h, which would remove carbonates, and then treated with 5 mL 0.5 mol L^{-1} $K_2Cr_2O_7$. The mixture was heated at 150–160 °C for 5 minutes, and then cooled at room temperature. The solution was transferred into a triangular flask with 100 mL deionized water. Unreacted $K_2Cr_2O_7$ was determined by titrating with 0.25 mol L^{-1} $FeSO_4$. Soil organic carbon content was calculated from the difference in $FeSO_4$ used between a blank and a soil solution without a recovery factor.

We used a pressure calcimeter method for the determination of SIC, which is similar to that of Sherrod et al. (2002). A subsample of 1.0 g soil was placed at the bottom of a 100-mL reaction vessel, then mixed with 2 mL 6 mol L^{-1} HCl containing 30 mg g^{-1} of $FeCl_2 \cdot 4H_2O$. After 2 h reaction, we measured CO_2 evolution in soil and standard samples by removing the aluminum tear off seal cap, which exposes the butyl rubber stopper, and inserting a 18-gauge hypodermic needle that was attached to the pressure transducer and voltage meter, and recorded the voltage reading to two decimal places after 3 to 5 seconds. A calibration curve was developed by mixing reagent grade $CaCO_3$ with oven-dried laboratory sand. Standard samples were made based on a final weight of 1.0000 g mixture of sand and $CaCO_3$ to obtain inorganic carbon concentrations of 1.2, 3.6, 6.0, 12.0, 18.0, 24.0, 30.0, 36.0, 42.0, 48.0, 54.0 and 60.0 g C kg^{-1}, respectively.

Loss-on-ignition (LOI) method was applied for estimating SOC and SIC. We used a programmable muffle furnace (S1849, Koyo Lindbeerg Co., Ltd, Japan) that has a capacity of combusting 32 soil samples at one time. First, 5 g soil was placed in a quartz glass and heated at 105 °C for 12 h to remove soil moisture, then the soil was combusted at 375 °C for 17 h (Wang et al., 1996). We calculated soil organic matter as the weight loss during combustion:

$$SOM_{LOI} = \left(\frac{W_{105\,°C} - W_{375\,°C}}{W_{105\,°C}} \right) \times 1\,000 \qquad (1)$$

where $W_{105\,°C}$ and $W_{375\,°C}$ are the soil weights after combustion at 105 and 375 °C, respectively.

Wang et al. (2011) reported that after combusting at 550 °C, combustion of sediments at 800 °C for 12 h is optimal for inorganic carbon content determinations. Thus, we carried out two more steps for SIC estimates, i.e., combusting at 550 °C for 2 h and then 800 °C for 12 h. We estimated SIC by the weight loss during combustion between 375 and 800 °C (LOI1), and during combustion between 550 and 800 °C (LOI2):

$$SIC_{LOI1} = \left(\frac{W_{375\,°C} - W_{800\,°C}}{W_{105\,°C}} \right) \times 0.273 \times 1\,000 \qquad (2)$$

$$SIC_{LOI2} = \left(\frac{W_{550\,°C} - W_{800\,°C}}{W_{105\,°C}} \right) \times 0.273 \times 1\,000 \qquad (3)$$

where $W_{800\,°C}$ and $W_{550\,°C}$ are the soil weights after combustion at 800 and 550 °C, respectively. The conversion constant of 0.273 in Eqs. 2 and 3 was used to convert the mass of CO_2 to the mass of C (i.e., 12/40).

Statistical analyses

Correlations and liner regressions were determined using Ferret Version 5.51 (NOAA's Pacific Marine Environmental Laboratory, USA; http://ferret.pmel.noaa.gov/Ferret/). Averages from the data were compared using Fisher's least significant difference at $P < 0.05$.

RESULTS AND DISCUSSION

Determination of SOC

To allow for a comparison between the LOI method and Walkley-Black method, a comparison in measured SOC by the automated analysis and the Walkley-Black method is shown in Fig. 1. There was a strong linear relationship ($r = 0.93$, $P < 0.001$) between the two methods. Because the intercept was not significantly from zero, the regression line was forced through the origin, which yielded a slope value of 0.99. Our results indicated that the oxidation of SOC by the Walkley-Black method was greater than 75% for most soil samples, with an average of 99%, which is much higher than some published values (less than 82%) for forest soils (De Vos et al., 2007), but in agreement with those (97%–102%) in most Tasmanian soils (Wang et al., 1996). The high recovery of SOC and large correlation

coefficient (0.93) in our study indicated that this modified Walkley-Black method could be used for measuring SOC in arid soils.
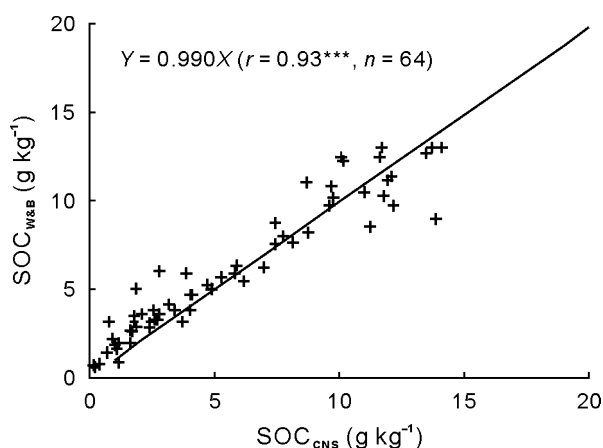

Fig. 1   Linear regression of measured soil organic carbon (SOC) between the automated analysis (SOC_{CNS}) and the Walkley-Black method ($SOC_{W\&B}$) for soil samples from set 2.

There was a strong linear relationship between the measured SOC by the automated analysis and the SOM determined by the LOI method, with a slope of 1.763 and an intercept of 4.37 (data not shown). The comparison suggested that the LOI method could provide good estimates for SOC in arid soils.

The 105 soil samples (set 2) collected in November 2010 were then used to compare SOC measured by the modified Walkley-Black method and SOM determined by the LOI method. The SOC content ranged from < 1 g C kg^{-1} to about 17 g C kg^{-1} by the Walkley-Black method, and SOM from < 1 to about 35 g C kg^{-1} by the LOI method (Fig. 2). There was a good linear relationship between the two methods:

$$SOC = 0.445SOM_{LOI} - 0.193 \qquad (4)$$

We used the soil samples from set 1 to evaluate the SOC-LOI relationship derived from soil samples in set 2. The comparison between the predicted SOC values with those determined by the modified Walkley-Black method showed that there was a strong regression ($r = 0.93$, $P < 0.001$) between them, with a slope value of 0.967 (Fig. 3). Table II illustrated that SOC was slightly overestimated by the LOI method using the Eq. 4. However, the bias, mean absolute error (MAE), and root mean squared error (RMSE) were small. Statistical analyses indicated that the differences were not significant.

Previous studies have shown various relationships between SOC and estimated SOM by LOI, which may be associated with soil properties and land use types
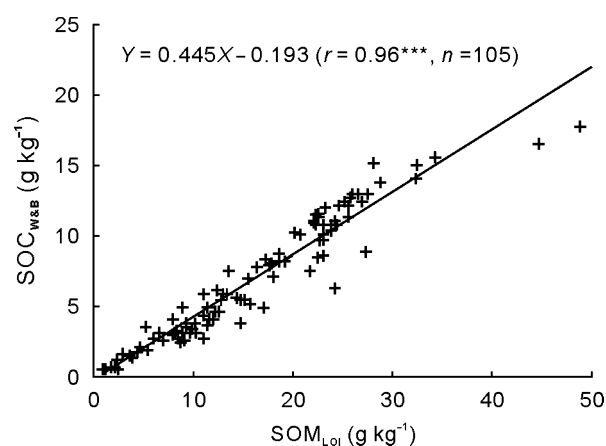

Fig. 2   Linear regression between measured soil organic carbon (SOC) by the Walkley-Black method ($SOC_{W\&B}$) and soil organic matter (SOM) by the loss-on-ignition method (SOM_{LOI}) for soil samples from set 2.
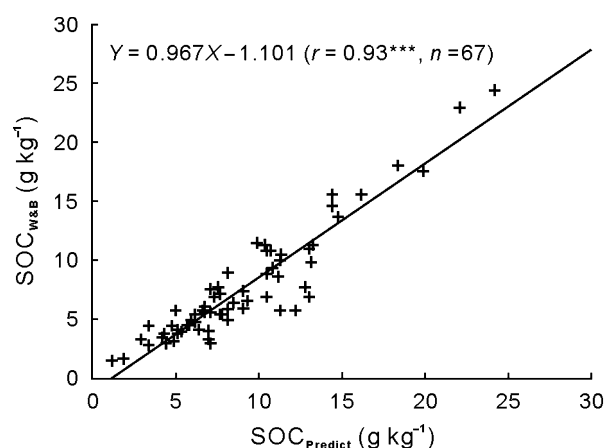

Fig. 3   Relationship for soil organic carbon (SOC) between the Walkley-Black method ($SOC_{W\&B}$) and prediction ($SOC_{Predict}$) using Eq. 4 for soils of set 1.

(Wang *et al.*, 1996; Konen *et al.*, 2002; Abella and Zimmer, 2007; Wang *et al.*, 2011). We found a conversion factor of 1.76 between SOM to SOC in the arid soils, which is close to the traditional value of 1.724 (Rowell, 1994), but smaller than the recent value of 2 by Pribyl (2010). While some reports (Chatterjee *et al.*, 2009; Szava-Kovats, 2009) suggested that the LOI method may overestimate SOM by causing some degree of dehydroxylation of clay minerals and/or oxidation of carbonate, our recent study indicated that overestimation mainly occurs in soils with SOC content of < 2.5 g C kg^{-1} (see Wang *et al.*, 2012). The uncertainty will be reduced when subsoil is excluded, and potential errors can be corrected using regression equations between a direct SOC measurement and the LOI method.

There have been many studies of using the LOI method to determine SOM/SOC in agricultural and forest soils, and sediments (Byers *et al.*, 1978; Heiri *et*

TABLE II

Measured soil organic carbon (SOC) by the Walkley-Black method ($SOC_{W\&B}$) and soil inorganic carbon (SIC) by the pressure calcimeter method (SIC_{PC}) for soils of set 1 and predicted SOC ($SOC_{Predict}$) and SIC using equations derived from soils of set 2 ($SIC_{Predict1}$ and $SIC_{Predict2}$)

Statistics	$SOC_{W\&B}$	$SOC_{Predict}$	SIC_{PC}	$SIC_{Predict1}$	$SIC_{Predict2}$
			g C kg^{-1}		
Maximum	27.71	31.43	47.22	47.34	46.61
Minimum	1.46	1.17	15.40	19.10	7.33
Mean	8.17	9.79	28.91	28.76	26.73
Standard deviation	5.65	6.08	6.18	5.64	7.42
Bias		1.62		−0.15	−2.18
Mean absolute error		1.70		1.22	2.55
Root mean squared error		2.23		1.52	3.59
t-value		0.85		−0.15	−1.87*

*Significant at $P < 0.05$.

al., 2001; Konen *et al.*, 2002; Campos, 2010; Salehi *et al.*, 2011). However, combination of temperature and duration varies widely, with temperature ranging from 150 to 900 °C and duration from 2 to 17 h in the literature (Wang *et al.*, 1996; Abella and Zimmer, 2007; Szava-Kovats, 2009). Reasonable results were obtained using either high temperature with short duration (Howard and Howard, 1990; De Vos *et al.*, 2005) or low temperature with long duration (Wang *et al.*, 1996; Konen *et al.*, 2002). Commonly, temperature ranges from 300 to 550 °C for determining SOC. This study demonstrated that LOI at 375 °C for 17 h should a good procedure for estimates of SOC in arid soils.

Determination of SIC

The SIC contents (about 10–50 g C kg^{-1}) were much higher than SOC contents in these soils (Table I). There was a strong liner relationship for SIC determined between the pressure calcimeter method and the LOI1 method using the soil samples from set 2 (Fig. 4):

$$SIC_{PC} = 0.942 SIC_{LOI1} - 2.877 \qquad (5)$$

We also obtain a good liner relationship between the pressure calcimeter method and the LOI2 method (see Fig. 5):

$$SIC_{PC} = 1.007 SIC_{LOI2} - 0.624 \qquad (6)$$

The LOI1 method produced smaller values for both the slope and intercept than the LOI2 method. Despite the considerable differences in the values of slope and intercept, both LOI methods revealed a strong correlation with the pressure calcimeter method ($r > 0.98$, $P < 0.001$).

The negative values of intercept by the LOI method indicated that there were non-SIC weight losses (*e.g.*, dehydration from other compounds) during combus-
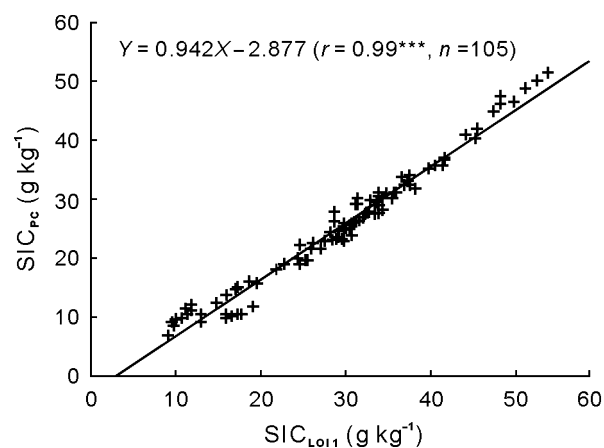

Fig. 4   Linear regression for soil inorganic carbon (SIC) determined between the loss-on-ignition method at 375 °C (SIC_{LOI1}) and the pressure calcimeter method (SIC_{PC}) using soil samples of set 2.
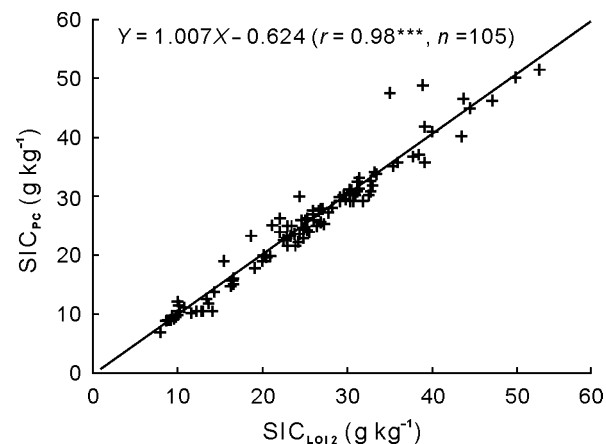

Fig. 5   Linear regression for soil inorganic carbon (SIC) determined between the loss-on-ignition method at 800 °C (SIC_{LOI2}) and the pressure calcimeter method (SIC_{PC}) using soil samples of set 2.

tion. Some researchers have showed that losses of lattice water in clay, gypsum, sulfide minerals, and metallic oxihydroxides may occur during heating process by

the LOI method, which leads to overestimates of carbonate (Allison and Black, 1965; Dean, 1974; Santisteban *et al.*, 2004).

Given that the LOI1 method yielded a smaller value for the intercept (about -3) than the LOI2 method (-0.6), one would predict that non-SIC weight losses would occur mainly during combustion between 375 to 550 °C, thus preferring the LOI2 method for estimating SIC contents. To evaluate the LOI2 approach, we used soil samples from set 1. Fig. 6 shows SIC values determined by the pressure calcimeter method *vs.* those predicted using Eq. 6. Surprisingly, most data points were placed above the 1:1 line although there was a good correlation ($r = 0.93$, $P < 0.001$) between the two methods. The slope value of 0.763 was significantly lower than 1, indicating some degree of uncertainty in using the LOI2 method to predict SIC contents. Some other studies have shown evidence of decomposition of inorganic C such as magnesite ($MgCO_3$), siderite ($FeCO_3$), rhodochrosite ($MnCO_3$) and dolomite [$CaMg(CO_3)_2$] during combustion at temperature between 425 and 520 °C (Heiri *et al.*, 2001; Santisteban *et al.*, 2004).
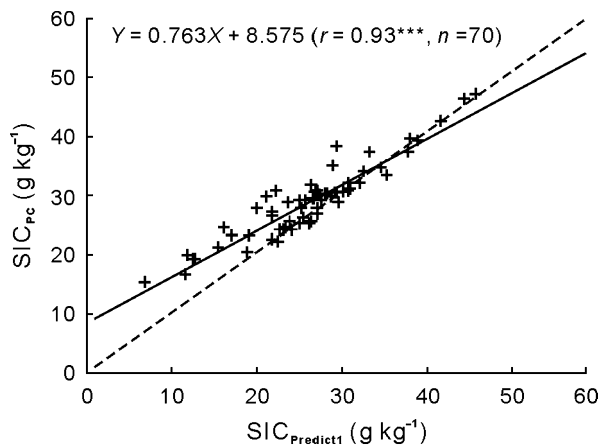

Fig. 6 Linear regression for soil inorganic carbon (SIC) between the pressure calcimeter method (SIC_{PC}) and prediction ($SIC_{Predict1}$) from Eq. 6 for soil samples from set 1.

In the present study, we evaluated the potential of the LOI1 method. As shown in Fig. 7, SIC values determined by the pressure calcimeter method and predicted using Eq. 5 were located along the 1:1 line. The LOI1 method showed a stronger correlation with the pressure calcimeter method ($r = 0.97$, $P < 0.001$) than the LOI2 method.

As shown in Table II, the LOI1 method produced a predicted SIC range of 19.1–47.3 g C kg^{-1}, a mean value of 28.8 g C kg^{-1} and standard deviation of 5.6 g C kg^{-1} for the soils tested, which were similar to those (15.4–47.2, 28.9, and 6.2 g C kg^{-1}, respectively) deter-
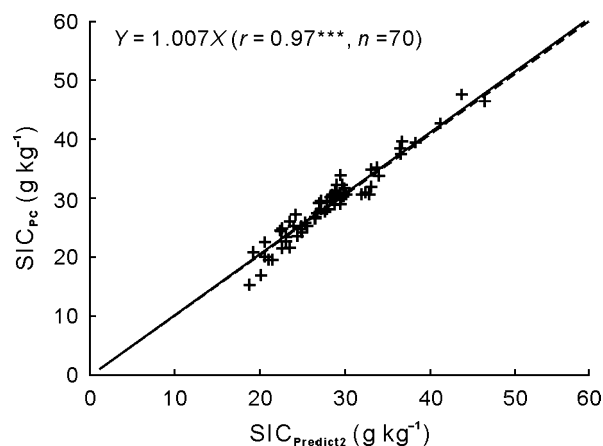

Fig. 7 Linear regression for soil inorganic carbon (SIC) between the pressure calcimeter method (SIC_{PC}) and prediction ($SIC_{Predict2}$) from Eq. 5 for soil samples from set 1.

mined by the pressure calcimeter technique. On the other hand, SIC predicted by the LOI2 method was significantly underestimated. These analyses indicated that there was no need to combust soils at 550 °C for SIC estimates, and a single step of heating soils at 800 °C, following the LOI procedure for SOC measurement, could provide accurate estimates of SIC for arid soils.

CONCLUSIONS

By comparing with the traditional methods for SOC (the Walkley-Black method) and SIC (the pressure calcimeter method), our study demonstrated that the LOI method was reliable and could provide accurate estimates of SOC and SIC for arid soils. To the best our knowledge, this is the first study to evaluate a two-step LOI approach for estimations of SOC and SIC, *i.e.*, 1) combustion at 375 °C for 17 h to estimate SOC, and 2) subsequent combustion at 800 °C for 12 h to estimate SIC. Future studies should be carried out to test the LOI technique in other parts of the arid and semi-arid regions.

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