Quantifying the carbon content of aeolian sediments: Which method should we use?

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A B S T R A C T

Total organic carbon (TOC) and inorganic carbon (carbonates) are important soil factors and paleoenvironmental proxies used worldwide. During past decades, several different methods have been proposed for determining these parameters from various sediment archives. Although the automatic elemental analyzer is gradually replacing the traditional quantification methods, it is critical to assess the reliability of all currently used methods to ensure accurate regional comparisons, carbon stock estimations and long-term carbon-cycle dynamics modelling. In this study, three most commonly used methods for determining TOC and carbonate content are compared using sediment samples from a sand-paleosol sequence located in northeastern China. Pearson's correlation between results of the different TOC and carbonate quantification methods produced high correlation coefficients, confirming substantial consistency among them. The automatic carbon analyzer yielded more accurate values with smaller uncertainties, while results of the general chemical method and loss-on-ignition (LOI) produced relatively reliable values with more data variability. Meanwhile, combustion at 500 °C to quantify the TOC content yielded significantly higher values than the automatic carbon analyzer and chemical methods, indicating that LOI method is not as reliable as previously thought. We attribute the overestimation to unsuitable SOM to TOC conversion factors and/or the presence of volatile minerals in the samples. In contrast with other studies, chemical wet oxidation of the sand-paleosol sediments did not significantly overestimate TOC, which commonly occurs in modern soils and lacustrine sediments. Our results suggest that the reliability of different methods for measuring carbon content could be affected by the sediment's origin and environment. Assessment of the results of the different TOC and carbonate quantification methods on additional types of sediment is needed so that researchers can use the most appropriate method for their study and make appropriate paleoenvironmental reconstructions given the sediments and methods used.

1. Introduction

Carbon content (including organic carbon and inorganic carbon/carbonates) is one of the most commonly used proxies for soil science (e.g. Vitti et al., 2016) and paleoenvironmental investigations (Dong et al., 2018). Employing TOC and TIC (carbonates) indices, numerous paleoclimatic studies have been carried out all over the world with the aim of investigating carbon cycles (Johnston et al., 2004; Chen et al., 2017) or to reconstruct past climate change (Shen et al., 2005; Long et al., 2010; Li et al., 2016). The TOC of aeolian sediments in arid and semi-arid lands, which is closely related to the paleovegetation density, has been used to reconstruct the paleoclimate and desertification history (e.g. Liu, 1985; Yang et al., 2008; Huang et al., 2009). Carbonates are also believed to be a reliable paoleoenvironmental indicator, and

have been used for regional paleoclimate studies (e.g. Diao and Wen, 1995; Wen et al., 1996; Yang et al., 2016; Zhao et al., 2017). Investigations of carbon dynamics in sedimentary records would greatly contribute to a better understanding of the interactions between carbon cycles and past climate change (Dean, 1999; Yu, 2011). An important first step is the precise and accurate determination of the carbon content of soils/sediments (Brye and Slaton, 2003; Vitti et al., 2016).

Different methods for determining the organic carbon and inorganic carbon content of soils have been developed (e.g. Walkley and Black, 1934; Dean, 1974; Heiri et al., 2001; Wright and Bailey, 2001). Among them, chemical methods (wet-oxidation for TOC and HCl neutralization for TIC), the loss-on-ignition (LOI, dry combustion) method and automatic carbon analyzer (e.g. elemental analyzer, EA) method are the most commonly used (Kristensen and Andersen, 1987; Boyle, 2004; Wang et al., 2012; Vitti et al., 2016; Yang et al., 2016). Several comparison studies have addressed the advantages and limitations of the different methods for different types of sediments (e.g. Wang et al., 2011, 2012; Vitti et al., 2016; Yang et al., 2016). For example, Wang et al. (2012) conducted a comparative study of chemical methods and the elemental analyzer method to determine the TOC and TIC content of lacustrine sediments, concluding that the wet-oxidation method could overestimate the TOC content when compared with the EA measurements. For the TIC measurement, the chemical methods and EA measurements displayed similar fluctuation trends (Wang et al., 2012). Few investigations, however, have considered aeolian sediments, which are widely distributed in arid and semi-arid China and used for paleoenvironmental investigations (Liu, 1985; Yang et al., 2011). Furthermore, recent studies have argued that the optimal quantification method could vary among different types of sediment (Wang et al., 2011; Kristl et al., 2016). Thus, an assessment of the reliability and consistency of different methods should be performed so that the most appropriate one for the circumstances is used.

Arid and semi-arid lands cover more than one third of China's mainland (Yang et al., 2011). Aeolian sediments, which are widely distributed in these areas, have been regarded as the principal regional paleoenvironmental archive (e.g. Liu, 1985; Yang et al., 2008, 2011; Huang et al., 2009). During past decades, all of the above-mentioned methods have been used to quantify the TOC and carbonate contents for aeolian sediments (e.g. Kemp et al., 1995; Yang et al., 2008; Huang et al., 2009). An assessment of the various methods is needed to facilitate further regional comparisons and to synthesize information from multiple investigations, given that previous data were obtained by different methods.

With the aim to assess and evaluate the reliability of different TOC and carbonate determination methods for use with sandy sediments, we present a TOC and $CaCO_3$ dataset from a sand-paleosol sequence located in Songnen sandy lands of northeastern China. Three different, commonly used methods were selected to independently quantify the TOC and carbonate contents of the sand-paleosol sequence. Further comparisons and assessments were performed on the data obtained to evaluate the advantages and limitations for each method. To facilitate future applications, several guidelines are proposed to help choose proper quantification methods for different sediments and different targets.

2. Materials and methods

2.1. Sample collection

The Daike sand-paleosol sequence (DK, 46°21′09″N, 123°21′43″E, 2.25 m in depth), located in Tailai County, Heilongjiang Province (Fig. S1), was sampled in the summer of 2015. The regional climate is strongly influenced by the East Asian monsoon system, which shows distinct seasonal variability. The mean annual precipitation is 397.2 mm while the mean annual temperature is 5.2 °C (Tailai station, from the year 1981 to 2010, http://data.cma.cn). The section contains

two paleosol layers (Fig. S2). The sandy layers are yellow and white whereas the paleosol layers are dark brown. In texture, the entire section is composed primarily of sand fractions, but with an increase in silt and clay content in the paleosol units. The section was sampled at 5 cm intervals throughout the 2.25 m exposure. A total of 41 samples were collected from section DK. All samples were oven-dried at 105 °C for 24 h to remove the moisture, then homogenized for further analysis.

2.2. Laboratory methods

2.2.1. Loss-on-ignition (LOI) method

The LOI method is based on the loss of mass caused by combustion of organic matter and decomposition of carbonates at high temperatures. For this analysis, oven-dried (105 °C) samples of approximately 4.000 g each were weighed in a crucible. Crucibles were placed in a muffle furnace (SX–8–10, Taisite, China) and combusted for 12 h at 500 °C. After combustion, the samples were cooled in desiccators to room temperature and weighed. The samples were then placed back into the muffle furnace and combusted at 800 °C for 12 h to determine weight loss. All samples were weighed with a 0.1 mg analytical balance (BSA124S–CW, Sartorius, Germany). The TOC and carbonate contents were calculated as the weight loss at 500 °C and 800 °C according to Eq. (1) and Eq. (2), respectively. To test the reproducibility of the process, during each processed batch, one in ten samples was analyzed three times to calculate the relative standard deviation (RSD) for that batch.

$$TOC_{LOI}\% = \frac{0.58 \times (m_{105} - m_{500})}{m_{105}} \times 100\% \qquad (1)$$

$$CaCO_{3LOI}\% = \frac{(m_{500} - m_{800})}{0.44 \times m_{105}} \times 100\% \qquad (2)$$

where m_{105} is the sample weight dried at 105 °C, while m_{500} and m_{800} are the sample weight after combustion at 500 °C and 800 °C. The 0.58 is the soil organic matter (SOM) to soil organic carbon (SOC) conversion factor (Pribyl, 2010) and the 0.44 is a conversion factor to transfer 800 °C weight loss to $CaCO_3$ content (Yang et al., 2016).

2.2.2. Wet-oxidation method for TOC measurements

The wet-oxidation (also known as Walkley-Black) method is based on the oxidation of organic C into CO_2 under a strong oxidant ($K_2Cr_2O_7$) in the presence of H_2SO_4 (Walkley and Black, 1934; Bao, 2000). For this method, subsamples of approximately 0.800 g of sediment were weighed and placed into test tubes. To each subsample was added 5 mL 0.5 mol/L $K_2Cr_2O_7$ and 5 mL concentrated H_2SO_4. The test tubes were then placed into an oil bath for 5 min at 185–190 °C to facilitate the oxidation of organic carbon. With 1,10-phenanthroline as a titration indicator, the unreacted $Cr_2O_7^{2-}$ was calculated by titrating with 0.2 mol/L $FeSO_4$ solutions (calibrated with standard $K_2Cr_2O_7$ solution). The carbon content was estimated from the $K_2Cr_2O_7$ volume consumed during the reaction. A coefficient of 1.1 was used for the oxidation efficiency (90%) calibration (Bao, 2000). Measurement errors were calculated by comparing results from three separate subsamples per field sample.

$$TOC_{WB}\% = \frac{C_{FeSO_4} \times (V_0 - V_1) \times 0.003 \times 1.1}{m} \times 100\% \qquad (3)$$

where C_{FeSO_4} is the molarity (in mol/L) of $FeSO_4$ solutions, V_0 is the volume (in mL) of $FeSO_4$ solution consumed by the blank, V_1 is the volume (in mL) of $FeSO_4$ solution consumed by the analyzed sample (in mL), the 1.1 is an oxidation efficiency calibration factor (Bao, 2000) and m is the dry weight of the analyzed sample.

2.2.3. Neutralization method for carbonate determinations

The neutralization method is based on the reaction of CO_3^{2-} with HCl, then neutralizing the excess HCl with the NaOH solution (Bao, 2000). For analysis, 25 mL 0.1 mol/L HCl solution was added to an

approximate 1.500 g subsample to dissolve the carbonates. Gently boiling the residuals to remove the CO_2 produced from the reaction. This step is essential because remaining CO_2 would consume the NaOH during the titrating, which would lead to an underestimation of the carbonate content. In the next step, the excess HCl was determined by titrating with 0.1 mol/L NaOH (calibrated with standard potassium hydrogen phthalate solution) under the presence of 3,3-Bis(4-hydroxyphenyl)-1(3H)-isobenzofuranone as the titration indicator. The carbonate content was determined from the volume of HCl consumed during the reaction according to Eq. (4). Reproducibility was estimated by including three replicates of the sample during each batch.

$$CaCO_{3HCl}\% = \frac{C_{NaOH} \times (V_0 - V_1) \times 0.05}{m} \times 100\% \quad (4)$$

where C_{NaOH} is the molarity (in mol/L) of NaOH solutions, V_0 is the volume (in mL) of NaOH solution consumed by the blank, V_1 is the volume (in mL) of NaOH consumed by the analyzed sample and m is the dry weight of the analyzed sample.

2.2.4. Automatic carbon analyzer

Total organic carbon (TOC) and carbonate content were also measured using a TOC analyzer with a solid sample module (Aurora 1030 W TOC Analyzer, OI Scientific). Two portions of each dry and homogenized sample (approximately 800 mg) were put into two individual small quartz boats after weighing, one for total carbon (TC) determination and the other decarbonized (carbonate free) sample for TOC measurements. The decarbonization was done with 1 M HCl for 12 h to remove the carbonates. Then all samples were combusted under O_2 at 900 °C, which oxidized the organic carbon and decomposed the inorganic carbon. The evolved CO_2 was carried to a nondispersive infrared (NDIR) CO_2 gas analyzer. The carbon content was calculated from the signals recorded by the NDIR detector. The TOC content of the decarbonized samples was obtained in the same way. The total inorganic carbon (TIC) content for each sample was calculated by subtracting TOC from TC. Then the carbonate content (expressed in $CaCO_3$) was calculated with Eq. (5).

$$CaCO_3\% = 8.333 \times (TC - TOC) \quad (5)$$

where TC is the total carbon content of the sample, TOC is the total organic carbon content of the sample and the 8.333 is a TIC to $CaCO_3$ conversion factor.

2.3. Statistical analysis

Pearson correlation was conducted to elucidate the relationships among the different methods. Using the least squares regression procedure, linear regression equations were fitted to the experimental data from different methods. Both the slope and intercept were calculated for the regression and the goodness of fit was evaluated through R^2. All the above analyses were conducted with R 3.5.0 (R Core team, 2018) with R Studio v1.1.463 (RStudio team, 2016).

3. Results

The TOC and carbonate measurement results reveal that the sand-paleosol sequence has a lower TOC content than the carbonate content (Fig. 1). The paleosol samples yielded more TOC and carbonates compared with sandy samples (Fig. 2). All data display very similar fluctuation characteristics (Fig. 1), however, the TOC contents determined with the LOI analysis are twice as large as those measured with the automatic carbon analyzer and wet-oxidation methods, suggesting an overestimate of the TOC content with the LOI method. For the carbonate profiles, all three methods show a consistent tendency and similar values, but with some slight differences (Fig. 1). The mean TOC content quantified by different methods varies from 0.307% to 0.872%, while the mean carbonate content (expressed as $CaCO_3$) ranges from 1.412%
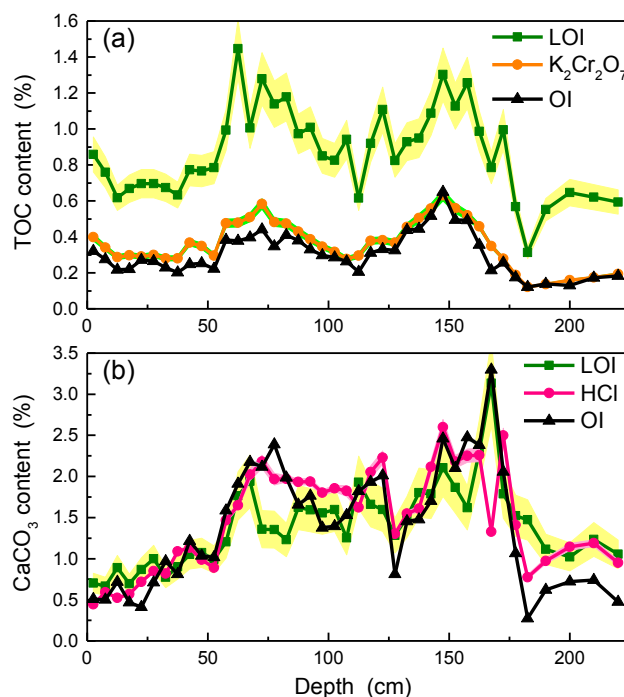

Fig. 1. TOC (a) and carbonate (b) content profiles of sand-paleosol section DK quantified using different methods.
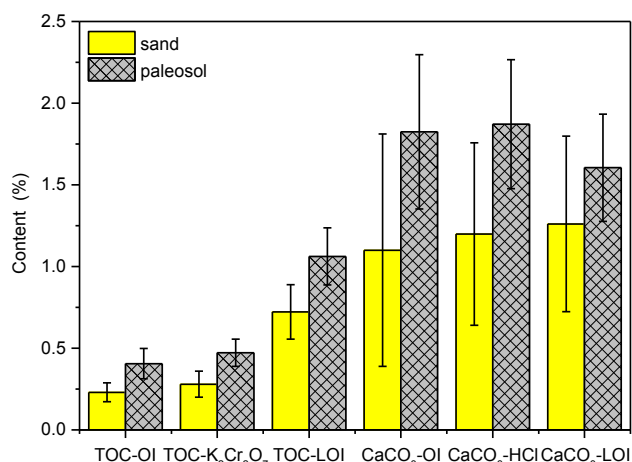

Fig. 2. Mean TOC and carbonate contents for the sandy layers (yellow bars) and paleosols (grey bars) obtained using different methods.

to 1.494% (Table 1).

4. Discussion

4.1. Comparisons of different TOC and carbonate determination methods

4.1.1. Comparisons of different TOC quantification methods

Pearson coefficients show that different TOC quantification methods correlate with each other better than the different carbonate results do (Table 2), indicating that the TOC profiles varied less than the carbonate profiles when using different methods. However, as shown in Fig. 1a, the LOI method clearly overestimated the TOC content of the aeolian sediments, despite its high Pearson correlation coefficient ($r = 0.848$, $p < 0.001$, $n = 41$). A regression analysis (Table 3 and Fig. 3) with a slope of 1.761 and an intercept of 0.331% ($R^2 = 0.720$, $n = 41$) clearly indicates that the LOI method yielded estimates that were about twice as high as those determined from the automatic

Table 1
Statistical descriptions of the TOC, carbonate data and standard errors quantified with different methods ($n = 41$).

		TOC-OI	TOC-$K_2Cr_2O_7$	TOC-LOI	$CaCO_3$-OI	$CaCO_3$-HCl	$CaCO_3$-LOI
Content	Minimum	0.121	0.122	0.313	0.274	0.446	0.665
	Mean	0.307	0.365	0.872	1.418	1.494	1.412
	Maximum	0.649	0.631	1.446	3.296	2.600	3.133
	SD	0.117	0.127	0.242	0.724	0.603	0.494
Measurement Error	Minimum	0.001	0.004	0.036	0.003	0.009	0.110
	Mean	0.003	0.017	0.099	0.016	0.043	0.234
	Maximum	0.006	0.031	0.164	0.032	0.093	0.519
	SD	0.001	0.007	0.027	0.008	0.025	0.082

TOC-OI denotes the TOC data were obtained with the OI automatic carbon analyzer; TOC-$K_2Cr_2O_7$ indicates the TOC data were obtained with wet-oxidation method; TOC-LOI denotes the TOC data were obtained with LOI analysis; $CaCO_3$-OI lists the carbonate data obtained with the OI automatic carbon analyzer; $CaCO_3$-HCl is the carbonate data obtained with the chemical neutralization method; $CaCO_3$-LOI denotes the carbonate data obtained with LOI analysis. All variables are in percentages (%) and SD is the standard deviation.

methods (Table 3).

Loss-on-ignition analysis is a rapid, relatively inexpensive approach for estimating organic carbon content (Wang et al., 2011). Assuming that the mass loss of samples is due to the combustion of organic matter, precise measurement requires an optimal temperature that is high enough to remove the organic matter completely, and at the same time low enough to minimize the mass loss caused by dehydroxylation of clay minerals and decomposition of carbonates (Sleutel et al., 2007; Pribyl, 2010; Kristl et al., 2016). Moreover, several non-carbon components in the sediments have been reported to cause an overestimation of organic carbon (e.g. Wang et al., 2011). For example, gypsum, sulfide minerals, and metallic oxihydroxides can impact the LOI analyses through oxidation, dehydration and decomposition under higher temperatures (Brauer et al., 2000; Rosen et al., 2002; Wang et al., 2011).

For aeolian sediments, several reasons could contribute to the overestimation of total organic carbon content with LOI analysis. One possible reason is that the empirical soil organic matter (SOM) to soil organic carbon (SOC) conversion factor of 58% used in Eq. (1), may not be applicable for aeolian sediments. Since LOI analysis measures weight loss directly, a conversion factor is always needed for translating organic matter content to TOC content. According to a critical review by Pribyl (2010), the conventional empirical factor of 1.724 (1.724 = 1/0.58) may be too low (Pribyl, 2010). In the present study, this is evidenced by comparisons between total carbon contents quantified by LOI analysis and OI automatic carbon analyzer. In Fig. S3, the TC content determined by LOI and carbon analyzer yield a linear regression of TC $_{LOI}$ = 1.393 × TC $_{OI}$ + 0.377%, R^2 = 0.875, p < 0.001, n = 41, suggesting an overestimation of TC content with LOI analysis. As the carbonate measurements of LOI do not yield significant overestimations, the TC overestimation mainly comes from the overestimated TOC content. In other words, the factor 58% may not be applicable to organic carbon estimates for our samples.

Regression between 500 °C combustion mass loss (in percentage) and TOC content determined by decarbonized samples yields a slope of 3.037 (Fig. S4), suggesting a TOC to OM conversion factor for our samples is about 32.93% (1/3.037 = 0.3293). Although this value could be revised with the addition of a new dataset in the future, the above analysis, at least, underscores the idea that a suitable conversion

Table 3
Regression parameters between different TOC and carbonate quantifications.

	Method	Slope	Intercept	R^2
TOC	$K_2Cr_2O_7$-OI	1.032	0.048	0.900
	LOI-OI	1.761	0.331	0.720
	LOI-$K_2Cr_2O_7$	1.661	0.266	0.757
Carbonates	HCl-OI	0.687	0.520	0.679
	LOI-OI	0.559	0.619	0.670
	LOI-HCl	0.552	0.587	0.454

The general regression model is $Y = aX + b$, where X and Y are TOC or carbonate content obtained with different methods. a is the slope and b is the intercept.

factor is required to guarantee the accuracy of LOI analysis.

Another possible reason for the overestimation of TOC with LOI analysis could be the existence of some volatile minerals in the samples. For example, in the Songnen sandy lands, soil alkalization has been reported as a major environmental problem (e.g. Wang et al., 2009). The accumulation of $NaHCO_3$ and Na_2CO_3 in soils is the main source of soil alkalization (Wang et al., 2009). Since $NaHCO_3$ has a very low decomposition temperature (Hartman et al., 2013), the decomposition of $NaHCO_3$ during the LOI measurement will lead to an overestimation of TOC content for sandy sediments (Boyle, 2004). In addition, the water of crystallization occluded in mineral crystals, such as gypsum, can increase the weight loss during combustion, which will lead to an overestimation of TOC content by LOI analysis. With multiple factors potentially influencing the accuracy of LOI data, those data should be treated with caution.

Interestingly, the TOC contents quantified by wet oxidation are very close to the values obtained by the automatic carbon analyzer (Fig. 1). This is quite different from previous studies. For example, in lacustrine sediments, there has been an obvious overestimation of the TOC content using the wet oxidation method (Wang et al., 2012). TOC contents estimated by the wet oxidation method in the present study correlated well with the data obtained from the automatic carbon analyzer (Table 3 and Fig. 3), having a slope of 1.032 and an intercept of 0.048% (R^2 = 0.900, n = 41). This suggests that these two methods of

Table 2
Pearson correlation coefficients of TOC and carbonate content determined with different methods ($n = 41$).

	TOC-OI	TOC-$K_2Cr_2O_7$	TOC-LOI	$CaCO_3$-OI	$CaCO_3$-HCl	$CaCO_3$-LOI
TOC-OI	1.000					
TOC-$K_2Cr_2O_7$	0.948[**]	1.000				
TOC-LOI	0.848[**]	0.870[**]	1.000			
$CaCO_3$-OI	0.592[**]	0.671[**]	0.731[**]	1.000		
$CaCO_3$-HCl	0.665[**]	0.624[**]	0.749[**]	0.824[**]	1.000	
$CaCO_3$-LOI	0.375[**]	0.398[**]	0.392[**]	0.818[**]	0.674[**]	1.000

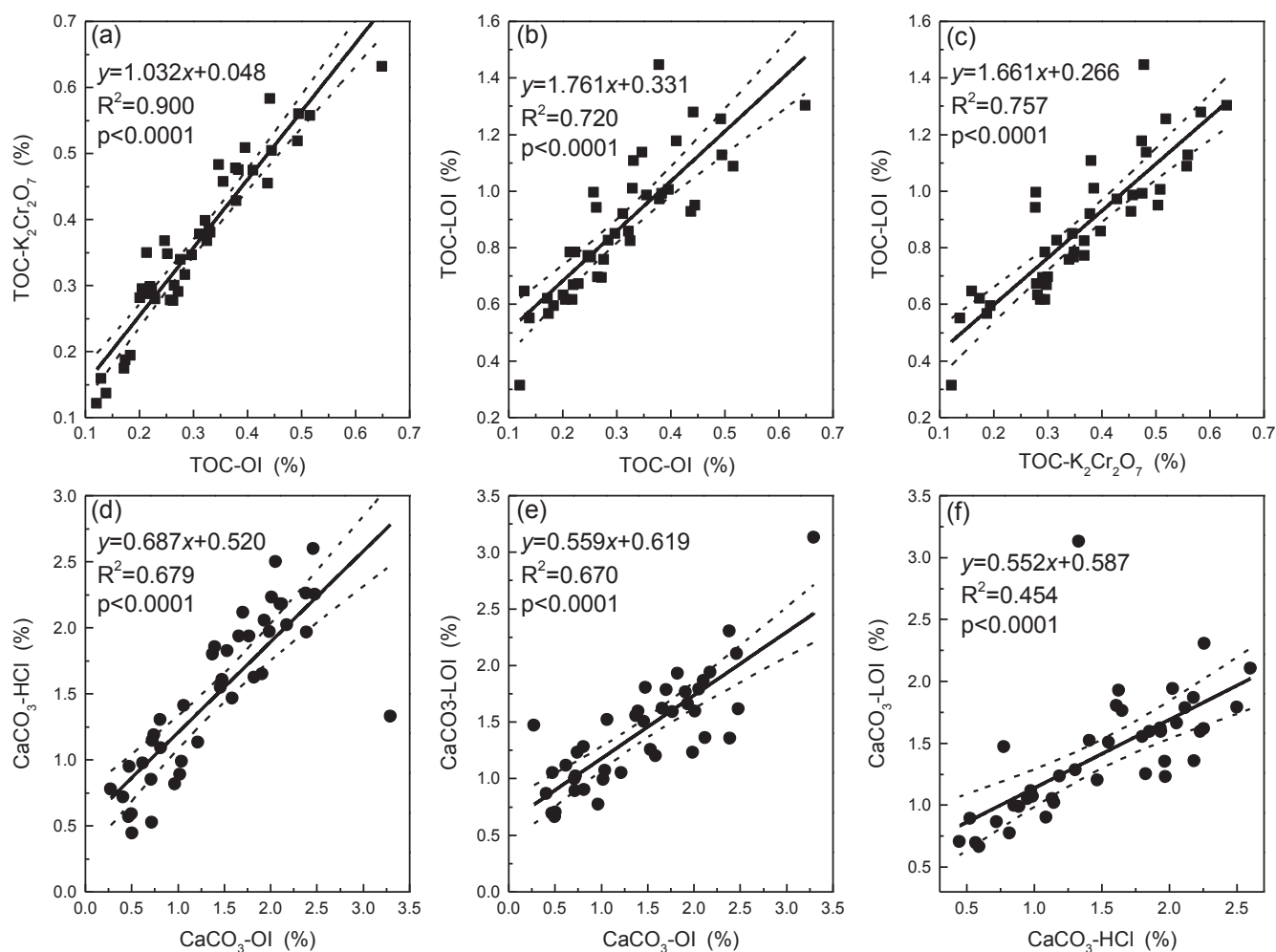[**] Statistically significant at $\alpha = 0.01$.

Fig. 3. Linear regressions between different TOC (a–c) and carbonate (d–f) quantifications.

determining the TOC content of sandy sediments are reliable (Figs. 1 and 3). Differences in the sediment origins should be responsible for the discrepancies between measurements in this study and those from previous investigations of lacustrine sediment. Wang et al. (2012) attributed the overestimation of TOC by the wet-oxidation method to the existence of reducing components (such as chloride, ferrous and manganese compounds) that would consume $K_2Cr_2O_7$. However, for sandy sediments in a dry climate always in an oxidative condition (Li et al., 2017), there are fewer reducing compounds to consume $K_2Cr_2O_7$. It is the organic carbon rather than reducing compounds that consume the $K_2Cr_2O_7$. Thus, for sandy sediments, both the wet oxidation and the elemental analyzer yield reliable measurements (Table 3).

4.1.2. Comparisons of different carbonate determination methods

Carbonate profiles for section DK quantified with different methods appear to be very similar to each other, except with sample 34, whose carbonate value determined with HCl neutralization is lower than that from the other two methods. The carbonate content determined by the carbon analyzer correlates well with that obtained with HCl neutralization and LOI methods (Pearson coefficient $r = 0.824$ and $r = 0.818$, respectively, $n = 41$). All three carbonate quantifications display very similar data ranges and errors (Figs. 1b and 4), underscoring the reliability of all three methods. Regression analysis between different methods show similar slopes and similar intercepts (Table 3 and Fig. 3). Even the LOI method yielded values that are not obviously biased, compared to the other two methods (Fig. 1b). The reason for this is that most of the vulnerable minerals or compounds are

decomposed during the TOC measurement (500 °C for 12 h). Calcium carbonate is quite stable below 600 °C but will decompose completely between 600 and 800 °C (Wang et al., 2011). Thus, with heating to 800 °C, almost all the weight loss of the samples comes from the decomposition of the calcium carbonates, which will make the LOI method yield carbonate content as reliable as the other two methods.

Overall, except for the overestimation of TOC with the LOI methods, these methods yielded consistent results, which is in agreement with previous assessments (Wang et al., 2011, 2012; Vitti et al., 2016). However, it should be noted that the LOI method could yield determinations with greater variability than the other two methods, as evidenced by the higher standard deviation values (Fig. 4), both for organic and inorganic carbon measurements (Fig. 4). The automatic carbon analyzer method provided more accurate values with the lowest measurement bias (Fig. 4). For sandy sediments, chemical methods (including the wet oxidation for TOC determination and HCl neutralization method for carbonates) provided relatively reliable values with moderate errors (Fig. 4), implying the reliability of the chemical quantifications.

4.2. Implications for further TOC and carbonates quantifications

Reliability, time-efficiency, cost (both equipment and/or chemicals) and environmental risks are the main factors to consider when choosing a specific technique (Lettens et al., 2007; Vitti et al., 2016). As summarized in Table S1, the automatic carbon analyzer can produce results in the shortest time. This allows for the analysis of more replicates,
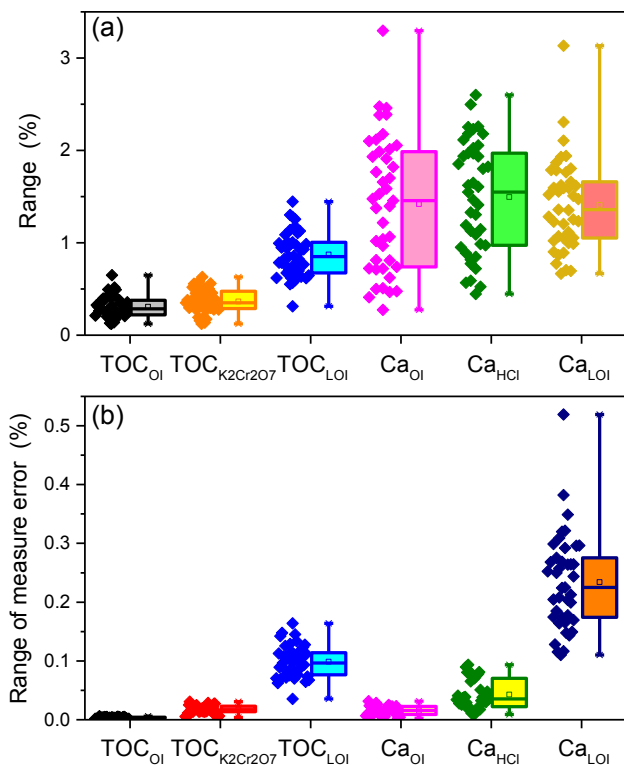
Fig. 4. Statistical parameters (in quantiles) of the TOC and carbonate data quantified with different methods (a); measurement errors for different TOC and carbonate quantification methods (b).

which will increase the precision of the results. Moreover, since the measurement is automatic, the constant presence of operators is not required. Finally, this method is safer than the others and environmentally friendly (Wang et al., 2012; Vitti et al., 2016). Using the LOI method to determine TOC and carbonate content has the advantages of simplicity, cost-effectiveness and no waste disposal (Wang et al., 2011). However, in this study, the LOI measurements yielded reliable carbonate content but overestimated the TOC content for sandy sediments, underscoring that a proper combustion temperature or suitable OM to TOC conversion factor is still needed for different sediments (e.g. Wang et al., 2011). The chemical methods can yield results of medium reliability (with smaller errors and standard deviations); their weaknesses are that they are time-consuming, require the constant presence of an operator and may be associated with environmental pollution (Vitti et al., 2016).

Results of this study indicate that both the wet oxidation and LOI methods could be affected by specific soil components, resulting in under- or overestimations of organic carbon content. The reliability of each method, therefore, likely varies with sediment type (Wang et al., 2011, 2012; Vitti et al., 2016). For example, Cl^-, Fe^{2+} and Mn oxides contained in a sample would lead to an overestimation of the TOC content (e.g., Wang et al., 2011, 2012; Vitti et al., 2016), which means that for lake sediments and peats, using a chemical method to quantify the TOC content could yield overestimated values.

Wang et al. (2011) analyzed samples with diverse sedimentary origins to discuss the optimal combustion temperature and time durations for using LOI to measure TOC or TIC content. They found that the weight loss (in %) for different samples varied with combustion temperature (Wang et al., 2011). In the present study, an unsuitable OM to TOC conversion factor and/or the presence of volatile minerals with a low decomposition temperature could lead to an overestimation for the TOC measurements (Fig. 1), and the more of those volatile minerals present, the larger the overestimation will be. It is important, therefore, to conduct additional comparison studies on samples from a variety of

sedimentary origins.

In summary, because all of the methods used in this study to determine TOC and TIC yielded similar profile trends for the sand-paleosol sediments, datasets obtained with the different methods can be compared directly with each other when the goal is paleoclimate reconstruction or regional comparisons. In those cases the trends displayed by the TOC (or carbonate) profiles are what is important, rather than exact values. However, for quantitative reconstructions or carbon cycle modelling, carbon content derived from an automatic carbon analyzer (or EA) is more efficient and preferable.

5. Conclusions

Understanding the reliability of various methods for determining organic and inorganic carbon content of aeolian sediments aids in interpreting and comparing datasets relevant to paleoenvironmental and paleoclimate reconstruction. Data from the three most commonly used methods for determining organic and inorganic carbon content were compared for a sand-paleosol sequence in northeastern China. Results indicate that, combusted at 500 °C, the LOI method could overestimate the TOC content of sandy sediments because of the presence of volatile minerals, for example, $NaHCO_3$, and/or use of an unsuitable OM to TOC conversion factor during the calculations. Contrary to results from previous work on lake sediments (Wang et al., 2012), in this study, the wet-oxidation method did not significantly overestimate TOC values for the sand-paleosol samples. This is attributed to the scarcity (or absence) of reducing constituents in the sand-paleosol sediments. Compared with chemical and LOI methods, the automatic carbon analyzer method yielded more reliable results with smaller measurement errors and higher precision. Carbonate measurements obtained through different methods showed similar trends for $CaCO_3$ profiles, suggesting that they all can track the inorganic carbon content well. An overall comparison of the advantages and shortcomings of each method highlights the importance of selecting the proper method to determine organic and inorganic carbon content for the investigation being conducted. Further studies should be undertaken to compare and evaluate results from the various methods on other types of sediment (or mineral composition) so that optimal methods can be determined for more sediment types.

Appendix A. Supplementary material

Supplementary data to this article can be found online at https://doi.org/10.1016/j.catena.2019.104276.

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