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1.2.3. Determination of Moisture Content and Total Organic Carbon within Basin Environments: Loss-on-Ignition

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ABSTRACT: Basin environments capture material from the surrounding catchment and preserve this within their sedimentology, enabling them to act as environmental archives. These archives provide geomorphologists with a wide range of proxy data from which to reconstruct palaeoenvironmental change. Of the available proxies, total organic carbon (TOC) remains one of the simplest to assess through loss-on-ignition (LOI). This technique adopts two phases; samples are initially dried and are then ignited at elevated temperatures to burn off all organic carbon, thus providing a measure of moisture and organic content respectively. The ease of assessment and links between organic content and climatic change make TOC an attractive proxy for researchers focusing on basin environments. Although the technique is simple, LOI has caveats which can lead to either under- or overestimation of TOC. This chapter provides an overview of the LOI process and highlights the potential issues which users could face when adopting the technique.

KEYWORDS: Loss-on-Ignition; organic; moisture; cores; basin environments

Introduction

Terrestrial basin environments (e.g. lakes and peat bogs) can act as rich archives when exploring palaeoenvironmental change. The ability of basins to preserve material captured from surrounding areas as stratigraphic profiles enables geomorphologists to use a wide range of proxy data to reconstruct palaeoenvironmental change. Despite likely variations in sedimentation rates, many basins will constantly accumulate material through background sedimentation (Gilli *et al.,* 2013); enabling them to produce continuous, high-resolution sequences. If sampled carefully, cores from these environments can produce records that are decadal or even sub-decadal in resolution (Battarbee, 2000).

The composition of basin deposits is particularly dependent upon the climate, as it can heavily influence both sediment supply and organic productivity (Leeder *et al.,* 1998; Ariztegui *et al.,* 2001). A large proportion of the organic material that is deposited into basins is derived from plant detritus (Meyers and Lallier-Vergès, 1999). Variations in the organic productivity and detrital availability of an area are typically recorded within basin profiles as fluctuations in Total Organic Carbon (TOC). The linkages between environmental conditions and the organic content of sediment (Meyers, 1997) make TOC an attractive proxy when reconstructing both abrupt and long-term climatic events from core samples.

Loss-on-Ignition (LOI) is a relatively quick and inexpensive method for determining the total organic carbon (TOC) throughout core lengths (Heiri *et al.,* 2001). Samples are initially dried and then held at high temperatures to ignite all present organics, the loss in mass at each of these stages providing an evaluation of moisture content (%) and TOC (%) respectively. Since LOI results are expressed as percentages it can be difficult to distinguish whether fluctuations are a result of absolute changes in organic or mineral matter (Birks and Birks, 2006), indicating that the technique is not suitable as an independent proxy. However, when used

in conjunction with other proxies, such as pollen (e.g. Beer *et al.,* 2007), LOI can provide valuable support when reconstructing palaeoenvironmental change.

Although this article addresses LOI for use upon terrestrial basin samples, the method is not restricted to this environment. The technique has also been used to assess forest surface soils (De Vos *et al.,* 2005), to identify sedimentological changes associated with tsunami phases (Hawkes *et al.,* 2007), and has been adopted in many other environmental settings. Regardless of the focus, studies adopting LOI will use the same, or a very similar, procedure to the one highlighted within this chapter (see Figure 2).

Despite being a relatively simple method, there are caveats which have led to the accuracy and precision of LOI being questioned within the literature (e.g. Santisteban *et al.,* 2004). But providing that the method is not used independently and that each stage is standardised, LOI remains a useful tool for geomorphological studies. This chapter provides an overview of the LOI technique and highlights some of the key decision-making aspects of the methodology.

Methodological Considerations

Sample size

There are various recommendations for how much sample to use for the LOI process, with some studies adopting given masses and others volumes. Although neither sample mass nor volume has been highlighted as superior measure for sampling, it is essential that sample size is always standardised. This is particularly important when dealing with organic rich samples and short ignition times (i.e. 2-4 hour) as increases in sample size have been shown to reduce the TOC estimated by LOI (Schulte *et al.,* 1991). If considering this factor, users may choose to use samples between 1 and 4 grams.

Ignition temperature

When selecting an ignition temperature it is essential to consider not just the combustion temperature of organics but the effects of these elevated temperatures upon the mineral fractions. Many peatland and lacustrine studies adopt a 2-4 hour 550°C ignition treatment for LOI (LOI_{550}) and commonly cite the methodology used by Heiri *et al.* (2001). However, the most suitable ignition time and temperature for samples can vary between core lengths, depending on their composition and sedimentary source (Wang *et al.* 2011).

Boyle (2004) highlights 4 simplified stages of thermal decay within organic matter (summarised in Table 1). On initial observation it would seem that an LOI_{550} treatment would be a logical protocol for assessing TOC, as the LOI_{550} combusts the most stable humified organics. However, it is clearly observed within the literature that clay minerals lose most of their structural water (dewater) above 450°C (De Vos *et al.,* 2005; Salehi *et al.,* 2011), meaning LOI_{550} would produce an overestimate of TOC should a sample be clay rich. Additional overestimation of TOC can be created through the loss of volatile salts, metal oxides and when using LOI temperatures of >500°C, through the loss of CO_2 from carbonates (Heiri *et al.,* 2001; Hirota and Szyper, 1975). As a result of this it has been suggested that the LOI_{550} should only be used for samples which are predominantly organic (Bhatti and Bauer, 2002) with only a small clay mineral fraction. Within the literature there appears to be no recommended organic to mineral ratio for ignition temperature selection. Users should therefore visually assess samples and consider their environmental setting, to determine the most likely composition of samples (i.e. organic-rich or mineral-rich).

Table 1. Stages of thermal organic decay (adapted from Boyle, 2004)

Stage	Temp (°C)	Process
1	40 - 135	Dehydration of plant matter
2	250 - 350	Breakdown of fatty acids, peptides and carbohydrates within organics
3	370 - 420	Decomposition of less stable humified organics
4	530 - 540	Decomposition of more stable humified organics

Many have adopted LOI procedures with lower ignition temperatures to avoid TOC overestimation created by clay minerals and carbonates. An example of this comes from Davies (1974) who tested the use of a 24 hour LOI treatment at 430°C and found excellent comparability between LOI_{430} results and the more traditional Walkley-Black method (Walkley and Black, 1934) for analysing organic content. The study also found that carbonates that were added to samples within the laboratory, were not impacted by the LOI_{430} procedure (Davies, 1974).

The method used by Davies (1974) adopts a long duration ignition which may not be suitable for processing a large quantity of samples, however, there are many variants of the LOI procedure with shorter ignition times that can be adopted (e.g. Table 2). Schulte and Hopkins (1996) and Schulte *et al.* (1991) provide an overview of various LOI ignition temperatures and durations adopted throughout the literature. They also highlight an R^2 value which highlights the comparability of the adopted LOI techniques with other more traditional methods for assessing TOC. Table 2 provides a selection of the ignition treatments highlighted within these studies.

Table 2. Example LOI treatments and comparability (R^2) with more traditional methods of obtaining TOC (adapted from Schulte and Hopkins, 1996; Schulte et al., 1991).

Temp (°C)	Ignition Duration (h)	R^2
360	2	0.97
400	8	0.97
430	24	0.99
500	4	0.87
600	6	0.86

N.B.: Not all data was used from original source. For full data tables see: Schulte and Hopkins (1996 - p23.) and Schulte et al. (1991- p.161).

When selecting an LOI treatment from the highlighted literature, it remains essential to consider the likely composition of samples, ensuring that those which are not organic rich are ignited below 500°C. The selected temperature should always be reported within any write-up (Heiri *et al.* 2001).

Most LOI methods are likely to either overestimate or underestimate TOC, owing to the overlapping combustion and dewatering temperatures highlighted above.

Crucible positioning

Although difficult to accommodate when processing a large number of samples, the positioning of crucibles within a furnace can impact the LOI results. Heiri *et al.* (2001) found differences of up to 3.7% between crucible furnace positions. Differences between crucibles positioned centrally and on the outer margins of a furnace were seen to be more significant with longer ignition periods (Figure 1). Despite the errors associated with crucible positioning appearing relatively small, they still contribute to the intrinsic uncertainties associated with the LOI technique and users should be aware of this potential error. When dealing with a small number of samples, users may choose to consult the results provided by Heiri *et al.* (2001) to minimise these errors by selecting the most reproducible positions within a furnace.

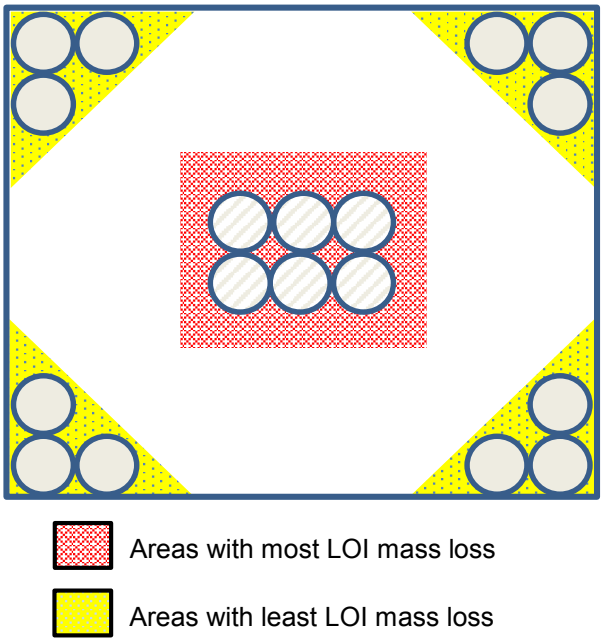

Figure 1. Plan view of crucible furnace positions and differences in LOI_{550} mass loss (adapted from Heiri et al., 2001).

Loss-On-Ignition Process

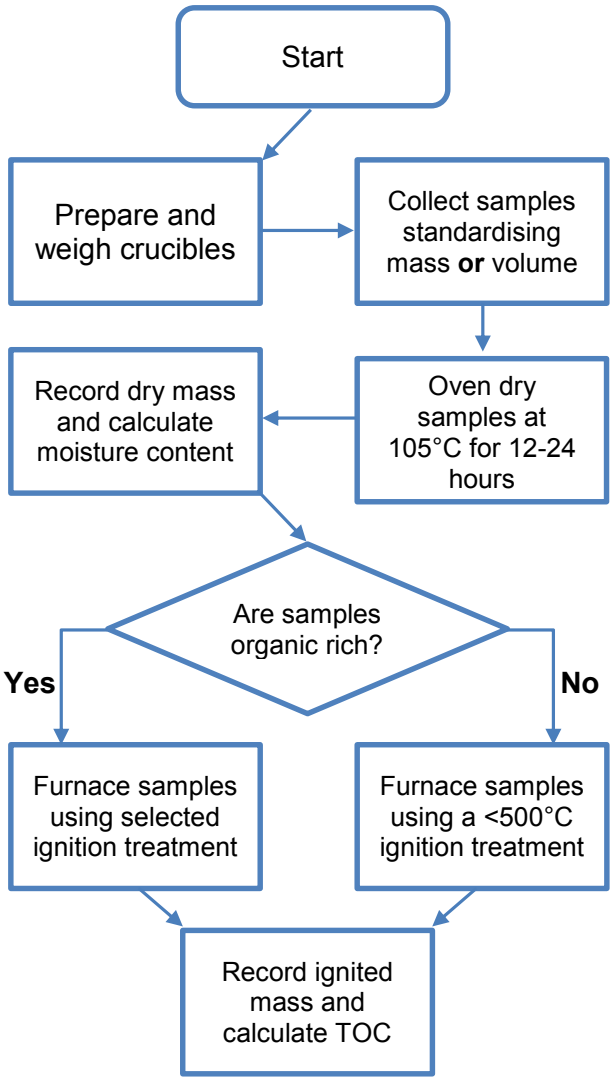

Figure 2. LOI procedure flow diagram.

Preparation

Porcelain crucibles are required for the LOI process as they can withstand the elevated temperatures used for ignition. Despite evidence suggesting that crucible volume does not impact LOI (Schulte *et al.,* 1991), it may be preferable to use the same size/shape crucibles to eradicate any potential inter-sample variations.

Prior to sampling for LOI, crucibles require some preparatory steps; a crucible for each sample, plus a few spares, should be prepared. New or soiled crucibles should be exposed to the selected ignition treatment prior to sampling, this ensures that crucibles can withstand the elevated temperatures and that all thermally unstable contaminants have been burnt off (Gale and Hoare, 1991). Following this initial stage crucibles can be labelled appropriately (e.g. depth of sample within core) using a permanent marker and weighed to at least three decimal places.

Figure 3. Obtaining crucible mass using a 4.d.p balance. Sampling spatula (left) and 1.25 cm^3 measuring spoon (right) included.

Core sampling

When dealing with samples of limited sample mass it may be preferable to sample volumetrically. This ensures that low density strata are not depleted when attempting to standardise sample size. For studies adopting a volumetric sampling strategy, it is common to use 1 cm^3 subsamples for LOI (e.g. Beer *et al.,* 2007; Dapples *et al.,* 2002; Shuman, 2003). However, if 1 cm^3 volumetric samplers are not available, there is the potential to use a spatula and 1.25 cm^3 ($\frac{1}{4}$ teaspoon) sampling spoons (Figure 3). These are affordable, easy to clean, produce replicable samples and are available from most homeware stores.

As core lengths will begin to dry out immediately upon opening, it is essential that LOI subsamples are collected swiftly, particularly if moisture content is required (e.g. for dose rate assessment in luminescence dating). It is recommended that crucibles are laid out on glass trays in numerical order prior to sampling, this helps users keep track when sampling and ensures that sample collection is as swift as possible. Before sampling material for LOI analysis the surface of cores should be removed, to

ensure that sample impacted by corer noise does not influence results. In this instance corer noise describes the contamination and distortion of stratigraphic units created during the coring process; this can be caused by frictional interactions between samples and the collection chamber. A spatula can be used to scrape away a thin layer of the unwanted material and, as with all core samples, users should scrape horizontally across cores to preserve their lithostratigraphic integrity.

Once the core surface is cleaned sampling can be conducted at the required resolution. Material should be removed from core tubing using a spatula and then placed into a measuring spoon. Note that compaction should be avoided, so material should not be pressed with force into sampling spoons. Collected LOI samples should be transferred from the measuring spoon into the prepared crucibles and weighed immediately. This provides the wet mass of samples.

Assessment of moisture content

Once the wet mass has been obtained, crucibles should be placed in an oven at 105°C for between 12 and 24 hours to dry samples (Gale and Hoare, 1991; Ben-Dor and Banin, 1989; Heiri *et al.* 2001). A 105°C oven treatment is adopted by a majority of studies as it will dehydrate most components within a sample. Also this drying temperature does not coincide with the ignition temperature of organics, meaning an underestimation of TOC will not occur during this process. After the drying period crucibles should be removed from the oven and immediately placed into desiccators to cool, this prevents samples from rehydrating. Once cool, samples can be weighed. An attempt should be made to restrict the amount of time the crucibles are exposed to the laboratory air to prevent rehydration. To calculate moisture content the following equation can be used:

$$Moisture\ Content\ (\%) = 100 \times \frac{WS - LOI_{105}}{WS}$$

Where: LOI_{105} and WS are dry sample mass (g) and wet sample mass (g) respectively.

Assessment of TOC

Key pieces of equipment are required for the ignition process (Figure 4). These include:

- Muffle furnace
- Tongs
- Heat proof gloves
- Safety Visor
- Heat proof trays

Figure 4. Furnace and safety equipment.

Before loading, users should preheat the furnace to the selected LOI temperature. When this temperature has been reached crucibles can be loaded using the tongs and safety equipment highlighted. Crucibles should be loaded into the furnace as a series of rows, to ensure that crucibles are easily identifiable. It is critical for users to map the position of samples within a furnace as the permanent marker may burn off.

Once the selected LOI treatment time has been achieved crucibles can be removed and placed onto heatproof tray. Samples should not be allowed to cool completely as they may begin to rehydrate, so should be relabelled and transferred into a desiccator (using tongs) prior to reweighing.

When the samples have been reweighed the TOC can be calculated using the following equation:

$$TOC(\%) = 100 \times \frac{LOI_{105} - LOI_{Ign.}}{LOI_{105}}$$

Where: LOI_{105} and $LOI_{Ign.}$ are dry sample mass (g) and ignited sample mass (g) respectively.

Further Uses

The environmental setting may influence the ignition temperature but it does not influence the LOI process and many studies using LOI will adopt a similar procedure to the one outlined here.

As an additional step, many studies employ an elevated 950°C ignition treatment following TOC acquisition, to assess the carbonate content of samples (Heiri *et al.* 2001). This provides an understanding of the minerogenic component of core samples. Carbonate content provides an additional aspect for multi-proxy studies and can be used to infer changes in a range of variables; salinity, catchment stability and climatically driven changes in mineral availability. (Cassina *et al.,* 2013; Birks and Birks, 2006).

Conclusion

This chapter provides an overview of the LOI technique and the associated methodological procedures. It is apparent that there is not an optimum ignition temperature for LOI and that users should take the composition of core lengths into consideration before selecting a thermal treatment. It is noted that LOI is not restricted to basins or core samples and that it can be adopted in many environmental settings. The overlapping dewatering and combustion temperatures of various materials mean that LOI will not provide a high precision estimate of organic or moisture content; however, if used in conjunction with other methods the results obtained from LOI can form a good basis for geomorphological interpretation.

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